ADVANCED DIRECT LIQUEFACTION CONCEPTS PROGRAM
PHASE II

RUN ALC-1 COAL CLEANING AND RECYCLE SOLVENT TREATMENT

Final Report

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EXECUTIVE SUMMARY

The DOE Advanced Direct Liquefaction Concepts Program was initiated in 1991 with the objective of promoting the development of new and emerging technology that has potential to reduce the cost of producing liquid fuels by direct coal liquefaction. The laboratory research program (Phase I) was completed in 1995 by CAER, CONSOL, Sandia and LDP Associates. A new three year bench scale test program was subsequently awarded in October 1995 to further develop several promising concepts derived from the laboratory program. The new program is composed of four continuous flow tests using a 2 Kg/h bench scale unit at Hydrocarbon Technologies, Inc. (HTI). The overall objectives of the first two tests are:

ALC-1	Coal Cleaning and Recycle Solvent Treatment
ALC-2	Advanced Slurry Catalysts

The first test ALC-1 was conducted between April 19 and May 14, 1996 and consisted of five test conditions over a period of 25 days. The feed coal was Wyoming sub-bituminous coal from Black Thunder Mine. Ash content of the feed coal was reduced from 5.5 to 2.3% (SO_3-free basis) by CONSOL using an oil agglomeration technique. The recycle solvent used in the last process condition was dewaxed and hydrogenated in a fixed bed reactor as an approach to quality improvement.

There were four main technical objectives for Run ALC-1:
1. To provide a baseline operating period with raw Black Thunder coal.
2. To demonstrate liquefaction of low ash coal produced by a low pH oil agglomeration technique.
3. To evaluate the impact of improving solvent quality by dewaxing and hydrotreating.
4. To operate under conditions leading to extinction of 343°C$^+$ material.

Improvements in liquefaction process performance were measured relative to previous results from Wilsonville pilot plant and HTI bench scale runs.

Run ALC-1 employed two slurry liquefaction reactors in series, an interstage separator, and a direct-coupled hydrotreater. An additional fixed bed reactor was used to hydrotreat the distillable (-524°C) portion of the dewaxed recycle solvent in Condition 5. Several recycle liquid and solid separation schemes were adopted throughout the run to minimize the lag time required to produce the desired recycle solvent and solids. The target total recycle and recycle solid to fresh feed ratio was 1.60 and 0.2, respectively. The removal of solids was achieved by pressure filtration. In Condition 1, in order to ensure a consistent oil content on the recycle solid, all pressure filtration cakes were toluene extracted. However, this caused considerable delay in establishing a steady state operation. Starting from Condition 2 onward, the lag time in the solid/liquid operation was significantly reduced by extracting only the net amount of cake that would be rejected. The portion of solid for recycle did not go through toluene extraction.

Two catalyst precursors were evaluated: Molyvan A and a HTI proprietary iron based gel. The

equivalent Mo and Fe content was 70-100 ppm and 0.7-1.0 w% of moisture-free coal, respectively. The precursors were premixed with the feed coal and recycle solvent before injecting them into a coiled preheater. Black Thunder coal used in this run was supplied by HTI and was the same batch that was employed in HTI's POC-02 run. The low ash oil agglomerated coal prepared by CONSOL, contained 3.3 w% ash (mf, SO_3-free basis), which was 40 w% lower than that of the raw coal.

The 25 day slurry catalyst operation went smoothly with no major mechanical or process problems. Excellent coal conversion of above 95 w% was obtained under all conditions. There were no handling problems with the agglomerated coal, it redispersed well in the recycle solvent. Also, oil agglomerated coal was more reactive with 3 w% higher coal conversion and increased distillate yield of 2-3 w% (maf coal basis).

Extinction of 343°C+ material was not achieved under the process conditions evaluated. However, at the low space velocity of 400 kg/h/m^3 reactor, the 454°C+ material was close to extinction (only 0.8 W% maf coal was produced). This fraction varied between 5.5 to 16.9 W% in other less severe process conditions.

The dewaxing and hydrotreating operation caused long delays in preparing distillate recycle solvent. Even at the end of 5th day of Condition 5, a solvent of steady quality was not obtained. For this reason, no definitive conclusion can be drawn regarding the impact of the dewaxing/hydrotreating operation on the process performance.

In view of the importance of achieving steady state operation conditions, it is recommended that the liquid/solid separation and solvent processing operation be simplified to shorten the turnaround time in future runs. Also, the practicality of achieving 343°C+ extinction should be examined and compared to high throughput but with some production of 454°C+ material.

BACKGROUND

The Advanced Concepts Program was initiated by the Department of Energy in 1991 to promote the development of new and emerging technologies that have potential, alone or in combination, to significantly reduce the cost of producing liquid fuels by direct coal liquefaction. Among other efforts, the advanced two-stage liquefaction technologies that were developed at Wilsonville over the past 10 years and by Hydrocarbon Technologies, Inc. have contributed significantly towards decreasing the cost of producing gasoline from coal. The approach has been to develop a long term strategy, in which promising liquefaction concepts, that have been identified mainly through fundamental studies, are subject to technical and economic evaluation on an increasing scale of experimentation for integration into a viable process configuration. The first phase of this strategy involved the laboratory-scale evaluation of selected concepts. The second phase will be concerned with evaluating the most promising concepts from Phase I in a continuous liquefaction unit. The outcomes of Phase II will be to ascertain the effects of scale-up on the projected improvements in liquefaction performance, and to provide a higher degree of confidence in the results and in the technical and economic assessments and forecasts.

OBJECTIVE

The objectives of Run ALC-1 were to demonstrate the liquefaction performance benefits of cleaning Black Thunder Mine coal, and the concept of solvent improvement through dewaxing and hydrogenation of the distillate portion of the recycle solvent. The coal cleaning method used was oil agglomeration at low pH. Expected benefits of coal cleaning include: higher coal conversion and higher distillate yield due to decreased organic rejection, and decreased dispersed catalyst requirement due to increased dispersed catalyst recycle. Potential benefits of solvent dewaxing and hydrotreating are improved coal conversion and improved resid conversion due to solvent quality improvements, lower gas make and decreased hydrogen consumption resulting from lower paraffin cracking, recovery of a valuable wax byproduct, and possible reduced severity resulting from the need for less cracking.

SCOPE OF WORK

The ALC-1 run plan included five run conditions that were selected to meet the technical objectives specified above. Interstage product separation was employed to reduce the yield of light gases (C_1-C_3) and to increase the residence time of 343°C+ fraction in the second reactor. In-line hydrotreating was conducted to improve product quality. Pressure filtration was used as the solid rejection device, allowing enough flexibility to recycle a high concentration of resid while maintaining a low resid-to-solid ratio in the rejected PFC. A vacuum still was also employed to separate VSB from PFL. The first condition was designed as a baseline operation and was carried out at a space velocity of 670 kg/h/m^3. Starting from Period 8 of Condition 2, extinction recycle of 343 °C+ VSB was attempted in order to maximize distillate yield. During Conditions 2 through 4, raw coal was replaced by oil agglomerated coal and space velocity, catalyst concentration, and ash content in the recycle stream were adjusted to improve distillate yield. Condition 5 was designed to study the effect of solvent dewaxing and hydrotreating on the process performance.

FEED MATERIALS

Raw and Low Ash Coal

The coal for this run was Wyoming sub-bituminous Black Thunder Mine. The pulverized coal was originally prepared by Empire Coke Co. for HTI's Run POC-02. A typical analysis of this batch of coal is given in Table 1. The moisture and ash content was 10.0 w% and 6.0 w% on dry basis, respectively. Its low sulfur content, 0.35 w%, was typical of Western sub-bituminous coal.

The low ash coal sample (590 Kg) was prepared ny CONSOL Inc. using a low pH oil agglomeration technique. The bridging oil was a heavy recycle distillate (V-1074) from the Wilsonville Pilot Plant end-of-run inventory from Run 263. Its analysis is shown in Table 2. Over 32.9 w% of this heavy distillate boiled above 454°C and the hydrogen content was 9.16 w%. Details of the preparation procedure can be found in a recent paper by CONSOL. As a percentage of the whole agglomerates (590 Kg), the agglomerates were composed of 7.6 w% moisture, 23.5 w% bridging oil, 2.3 w% SO_3-free ash and 66.7 w% maf coal. Composition of each individual batch is presented in Table 3. At low pH there was significant reduction in the sodium, calcium and magnesium content of the ash, as shown in Table 4.

Startup and Makeup Oil

L-814, which was the end-of-run makeup oil in Tank #4 from HTI's POC-02 run, was used as startup oil to bring the unit to near operating conditions and was the initial vehicle oil for slurrying the coal for Condition 1. As process derived solvent was avaliable, L-814 was phased out gradually. L-814 contained a unknown proportion of coal and petroleum (hydrotreated FCC decant oil) derived materials. It has a low API gravity of 0.4° and over 40.5 w% of 454°C$^+$ materials (Table 5). Apart from the first couple of days, L-814 was not required in the subsequent periods.

Dewaxed and Hydrogenated Distillate

About 200 kg of DW-HT VSOH was prepared prior to the Run ALC-1. The starting material VSOH was obtained from previous bench runs (CMSL 9-11, PB01 and 02). Their analysis is shown in Table 6.

Catalysts

A dispersed dual slurry catalyst system composed of Molyvan A and HTI iron based gel catalyst was used in the entire run. On a dry coal feed basis, molybdenum and iron concentrations were 70-100 ppm and 0.7-1.0 w%, respectively. Sulfur additive was incorporated into each stage to provide an equivalent of 3.0 w% of sulfur in each of the reactor.

The two fixed bed hydrotreaters were packed with Criteron C-411 trilobe catalyst.

SYSTEM CONFIGURATION

HTI's two-stage bench scale unit was reconfigurated to meet the specific requirements of ALC-1.

The major components for ALC-1 are:

- a 31.8 liter feed tank (P-2) and two parallel charge pumps (J-1 & J-2)
- a 1330 cc coiled preheater
- a 2000 cc first stage backmixed reactor (K-1)
- a first stage product recovery system (O-1A, Hot Separator; O-2A Cold Separator)
- a 2000 cc second stage backmixed reactor (K-2)
- a second stage Hot Separator (O-1)
- a 500 cc fixed-bed direct coupled hydrotreater
- a second stage product recovery system:
    - O-2 Cold Separator
    - O-3 Hot Separator Bottom Flasher
    - 12" pressure filter
    - vacuum stills
    - acetone dewaxing and solvent recovery unit
    - filter cake toluene extraction unit
- Two 250 cc fixed-bed off-line solvent hydrogentaion unit

Liquefaction Unit (HTI Unit 227) Configuration

A schematic flow diagram is depicted in Figure 1. Coal or agglomerated coal was premixed with catalysts, sulfur additive, recycle solvent and solids in a small batch prior to charging to the Feed Tank (P-2). Joined by feed hydrogen, the gas/feed slurry stream passed through a short residence time coiled preheater (5 minutes). Temperature of the feed mixture was raised to 140°C below the first stage reactor temperature. The preheated feed mixture enetered the bottom of the first reactor and exited through the top. An internal circulating pump returned a portion of the reactor slurry to the bottom of the reactor providing the backmixing action. The first stage light reaction product was cooled and separated from the main slurry. This light product, along with other second stage non-hydrotreated products, were fed to the direct-coupled hydrotreater. The main slurry was further liquefied in the second stage backmixed reactor.

The hot vapor products from the second stage liquefaction reactor were separated in the hot separator and fed directly into a fixed bed hydrotreater. The hydrotreater (K-3) was connected directely with the hot separator without pressure reduction. Hydrogen, C_1-C_3 hydrocarbons, heteroatom gaseous products, water and volatile liquid products from the overhead of the separator passed through a mixing phase trickled bed hydrotreater. The main function of the hydrotreater was to stabilize the hydrocarbon products and to reduce heteroatoms (N,S,and O). The hydrotrerater was packed with 500 cc Criterion C-411 3mm trilobe catalyst. Temperture of K-3 was controlled at 379°C throughout the entire run.

In addition to the hot vapor from the separator, several other light product streams were also fed to the hydrotreater. These streams were first stage overheads (water-free), atmospheric flash from the hot separator bottoms, atmospheric still overheads and all other unit knockouts. These materials were blended and injected using a single pump.

Solvent Preparation and Solid Rejection

The heavy liquid/solid separation system consisted of the following components: Atmospheric Still, Pressure Filtration, Vacuum Still and Toluene Extraction unit.

The solid containing heavy product stream from the bottom of hot separator was flashed, then distilled in a packed atmospheric still. The topped product slurry was filtered under elevated pressure and temperature. The solid-free liquid constituted the recycle solvent, while the oil-laden filter cake was further extracted with toluene to reduce the amount of organic rejection.

The atmospheric still operation experienced bumping problems during the first couple of days of the run. The packed bed was clogged with solids and became ineffective in providing the proper cut points. High boiling materials and possibly some unreacted coal ended up in the direct-coupled hydrotreater (K-3). The still was bypassed from Period 5 onward.

At the beginning of the run, all filter cake was extracted with toluene to control the oil content of the recycle solid and to minimize the amount of oil rejected with the solid residuals. However, the extraction and toluene recovering operation caused significant delay in establishing a steady state operation. Several improvements were subsequently implemented to shorten the recycle solvent and solid processing time. One of these improvements was to recycle the oil-laden filter cake without toluene extraction. Also, a light distilate cut from the vacuum still operation was used as a diluent to reduce the viscosity of the feed to the filter. The recycle solvent and solid recovery schemes used in the run are illustrated in Figures 2 through 5.

Solvent Dewaxing and Hydrogenation System

In Condition 5, the distillable portion of the recycle solvent was dewaxed using acetone at a reduced temperature of -10°C. The dewaxed solvent was subsequently hydrotreated to improve its hydrogen content. Dewaxing was performed in a batch mode, as shown in Figure 6. About 18 kg of 524°C- cut from the vacuum still was charged to a mixing tank to which acetone, in a ratio of 3/1 (acetone/feed), was added. The mixture was agitated for about 60 minutes minutes and then transferred to a jacketed chiller. Temperature of the mixture was brought down to -5°C. Sufficient time (120 min) was allowed to ensure proper precipitation of the waxy material. The wax was recoved by using two filters (25 and 2 micron) connected in series. Acetone was separated from the dewaxed distillate by flashing at atmospheric temperature.

The dewaxed distillate was then fed to a fixed-bed hydrotreater. The hydrotreater was made of 0.957 inch diameter tube with a catalyst loading of 253 cc. The reactor tube was immersed in

fluidized sand bath heater and was maintained at a temperature of 360°C, with a system back pressure of 15 MPa. The feed rate was approximately 650g/hr. Hydrogen was fed at a rate of 0.652 m^3/hr in each reactor. Two reactors were used for hydrotreating.

RUN PLAN

Run ALC-1 consisted of five process conditions extending over 25 periods(days). As shown in Table 7, Condition 1 (Periods 1-6) was a baseline condition designed to compare with HTI's CMSL-9 Condition 6, but using Molyvan A and HTI Fe gel catalyst, instead of HTI Fe powder catalyst. Temperatures of the major reaction vessels were held constant throughout the entire run:

Preheater:	300°C
1st stage Reactor:	440°C
2nd stage Reactor:	450°C
In-line Hydrotreater:	379°C
1st stage Hot Separator	332°C
2nd stage Hot Separator:	340°C

Also, with the exception of Condition 5, the ratio of total recycle to moisture free coal was 1.60. When oil agglomerated coal was used, Conditions 2 through 4, the bridging oil on the agglomerates was considered as recycle solvent. In each case, the amount of process derived solvent for recycling was reduced accordingly to maintain a recycle/feed ratio of 1.60. Fresh molybdenum and iron contents were 100 ppm and 1.0 W% of mf coal. The mf coal space velocity was 670 kg/h/m^3 in each reactor. Solid recycle was achieved by recycling toluene-washed PFC. The recycle stream consisted of pressure filter liquid (PFL) and a portion of the toluene-extracted PFC. The vacuum still was not used for Condition 1. The recycle strategy was to satisfy the recycle solid concentration (0.2 recycle to mf feed). Once this target was attained, the excess toluene washed PFC was rejected. The balance of the recycle consisted of PFL.

Due to the long cycle time (12 hours) for PFC extraction with toluene, the target recycle solid ratio was not achieved until the fourth day of operation. As a result, Condition 1 was extended by one period and completed after Period 6. Also, Condition 1 was not operated in the resid extinction mode. There was an excess of PFL (approximately 18 W% mf coal), which was credited as product. Toward the end of Condition 1, the PFC extraction time for recovering oil on the cake was shortened from 12 hours to 6 hours.

Starting from Condition 2 (Periods 7 to 13), the feed was switched from raw coal to a low ash , oil agglomerated coal. Also, early in this condition, the recycle strategy, as well as the space velocity, was modified aiming at extincting the 343°C+ material. Firstly, the space velocity was decreased from 670 to 560 kg mf coal/h/m^3. Secondly, all PFL was sent to the vacuum still, and all the vacuum still bottoms(VSB) and enough of the vacuum still overhead (VSOH) were recycled as necessary to satisfy the total recycle requirement. Any extra VSOH (413°C-) material, which showed approximately equal amount to the distillate agglomerating oil, was collected as product.

In Condition 3 (Periods 14-17), the make-up catalyst rate was reduced by 30 % relative to Conditions 1 and 2, while other process parameters remained unchanged. In order to minimize the recycle of

light material, a second vacuum still cut point of 343°C was implemented. The 343-423°C stream became the source of agglomerating oil equivalent and recycle distillate.

In Condition 4 (Periods 18-20), all process parameters were the same as in Condition 3, except that the space velocity was further reduced to 400 kg mf coal/h/m^3, so that all 343°C+ could be extincted.

In Condition 5 (Periods 21-25), the feed was switched back to raw Black Thunder coal. In order to reflect any possible process improvement as a result of the higher quality recycle solvent(dewaxing and hydrotreating), it was decided to keep the catalyst make-up rate at 70% of the Condition 1 value, and to set the space velocity at 480 kg mf coal/h/m^3 reactor. The entire PFL stream was vacuum distilled at 524°C to produce 524°C- VSOH for dewaxing and hydrotreating. Waxes from the Ketone dewaxing unit was kept as a product stream. The dewaxed and hydrogenated VSOH was fractionated and the 343°C+ portion was recycled. Due to the long turnaround time for dewaxing and hydrotreating operation, an initial batch of DW-HT distillate was prepared using Black Thunder coal-derived liquid from previous HTI bench runs. This material constituted a large percentage of the recycle solvent during the early part of Condition 5, and was gradually replaced by process produced DW-HT distillate in the last three days of Condition 5.

DATA MANIPULATION

Feed Streams

The spread sheet program provides for 5 fresh feed streams, 5 non-fresh feed streams and 10 product/recycle streams. Each stream type is handled differently in the calculations of yield. The 5 fresh feed streams (normally coal, feed oil, waste plastic, etc...) have not yet passed through the reactor and are considered the fresh feed to the system. The net product slate is divided by only the coal stream to determine W% of feed coal. None of the other 5 fresh feed streams were used for this run. The 10 product/recycle streams (VSOH, PFL, ASOH, VSB, PFC, etc...) are entered both as feed streams and as product streams. The net for each stream is calculated and used to determine the yields. The 5 non-fresh feed streams (makeup oil, dispersed catalyst, agglomerating oil) are considered as neither product/recycle nor fresh feed streams. They are considered as inert and simply pass through the system unchanged. The analysis for these streams is subtracted from the product slate as if they did not exist. If these streams are changed in the reaction then the analysis that would need to be entered for them would be the analysis of the products that they form. For this run the only fresh feed stream that is used is the coal. All oil streams are handled as product/recycle streams or as non-fresh feed streams.

Gas Analysis

Two gas streams are provided in the program. These gas streams handle nitrogen and oxygen content differently due to the different streams from the unit. The vent stream should contain no nitrogen or oxygen, just hydrogen and hydrocarbon gases. All nitrogen and oxygen in these streams is considered as a contaminant from air and eliminated from the analysis and the analysis of the gas fractions is re-normalized to 100% and then applied to the entered gas flow. The bottoms gas streams should contain nitrogen (from purges) but no oxygen. The oxygen is eliminated from the analysis along with a proportional amount of N (79/21) as if the contaminant was air and then the analysis of the gas fractions is re-normalized to 100% and applied to the entered gas flow.

Normalization Factor

The normalization factor was changed from one based upon total material recovery to one based upon carbon balance. The carbon content of all feed streams and product streams is calculated to equal 100% carbon balance. The normalization factor is applied to all gross (not net) product streams in order to achieve this. The normalized gross products are then used to calculate the net products and then the product fraction yields.

The addition of TNPS to the feed affect the normalized yield. According to a microautoclave test under coal liquefaction conditions, TNPS was decomposed to release H_2S and light hydrocarbon gases. It was found that only 12 W% of hydrocarbon portion of TNPS was decomposed to form gaseous products (majority is C_1-C_3) and the remaining 88 W% was essentially light liquid with a boiling range of IBP-343°C. As a result, in the normalized yield calculation, the hydrocarbon portion of TNPS was backed out from corresponding gas and liquid products.

Sulfur and Nitrogen Balance

The sulfur and nitrogen balance are calculated and the difference is assumed to be the H_2S and NH_3 product. This calculation forces these elements to balance. The sulfur present in the feed H_2S and the TNPS is not included in these calculations, so the results will only reflect the H_2S and NH_3 produced from coal.

Desulfurization and Denitrogenation

The quantity of sulfur/nitrogen fed is defined as the sulfur/nitrogen from the coal (organic+inorganic). The quantity of sulfur in the products is defined as the net (product out- recycle fed in) for each of the product/recycle streams, except for PFC. The PFC is not included in the product sulfur/nitrogen calculations. The sulfur present in the H_2S and the TNPS is not included in these calculations so that the results will only reflect sulfur/nitrogen removal from the coal.

Ash Balance

The total ash in the fresh feed is calculated. The quantity of ash present in the PFC is also calculated based upon actual collected PFC and PFC analysis. The quantity of PFC collected is then adjusted to achieve a 100% ash balance. The opposite of the adjustment that was applied to the PFC is applied to the PFL and its products (VSOH, VSB etc...). For example, if the PFC was adjusted down by 250 gms, then the PFL would be adjusted upward by 250 gms (or possibly the total of the VSOH and VSB would be adjusted upward by 250 gms, proportional to how they were produced from the PFL). This maintains the same material recovery as well as balancing the total ash.

PFC Oil Content

The oil content of the PFC is assumed to have the same analysis as the oil in the PFL. However, for those periods where there is no PFL analysis, the TGA analysis of the PFC is used to calculate a 524°C- and a 524°C+ content.

Coal Conversion

Coal conversion is calculated on both a straight basis and on a SO_3 free basis. The SO_3 free basis is calculated by assuming that the sulfur content of the ash is in the form of SO_3 which is eliminated from the total ash content. This adjustment is made to both the feed ash and the product ash. The option is given of which method to use for purposes of calculating and reporting yields and conversion.

Water Yields and Oxygen Balance

Oxygen balance is calculated and the water yield is adjusted to achieve 100% oxygen balance.

First Stage Coal and Resid Conversion

The first stage slurry sample was separated into PFL and PFC by pressure filtration. The QI and ash contents of PFC and resid content of PFL were analyzed. The total ash in the feed streams (coal, recycle streams and catalyst) is calculated and the quantity of PFC is adjusted to achieve 100 % ash balance. The IOM in adjusted PFC represents unconverted coal IOM and the IOM "carried over" from recycle PFC. The IOM in recycle PFC is considered to remain unchanged through K-1. In general, the first stage coal conversion is calculated on the basis of maf coal only.

The resid conversion is defined as ((Resid In-Resid Out)/Resid In)*100. The "Resid In" is the total resid in the feed streams including coal IOM and resid from recycle streams while "Resid Out" is the total resid in the first stage slurry calculated through ash balance.

RESULTS AND DISCUSSIONS

Overall Process Performance

In view of the complexity of the unit operations, especially the liquid/solid separation system, the overall material balance for each operating day was exceptionally good. As shown in Table 8, material balance of most periods was between 96-100%, except that of Periods 4,14,18,20 and 21. Among these periods, only Period 20 was the last day of a process condition. As a result, results from Period 20 should be viewed with caution.

In Condition 5, the foreign DW-HT distillate was not completely replaced with process derived distillate even after five days into the condition. However, the foreign DW-HT distillate was produced from the same coal and used an all dispersed catalyst system. The use of non-steady solvent quality should not have a significant impact on the outcome of the test conditions.

Detailed material balance data from the 25 day operation is presented in Appendix A to E. The total recycle and recycle solid to MF coal feed ratio was within ± 0.1 and ±2.1 of the target values, respectively (Table 8).

Two Stage Performance

Coal Conversion

Coal conversions reported in Table 10 and Appendices A to E are on an SO_3-free basis, which serves as a common basis for comparing the raw coal and the low ash (low calcium) oil-agglomerated coal. Using an all dispersed catalyst system, coal conversion for all conditions varied from 93.4 to 98.7 W%.

During the oil agglomerated coal conditions (Conditions 2 to 4), coal conversion was consistently higher (3 W% higher than raw coal conditions), indicating that oil agglomeration improves coal conversion, presumably because of reduced organic rejection. From Condition 2 to Condition 3, the same space velocity of 561 kg/h/m^3 was maintained while the catalyst concentration was reduced by 30 W%. The reduction in make-up catalyst concentration didn't affect coal conversion. It should be noted that from Condition 2 to Condition 3, the ratio of ash/MF coal in the recycle stream increased from 13.9 W% to 14.7 W%, implying that slightly more spent catalyst was added to the recycle stream.

Coal conversion obtained from Condition 5 may be compared with that from Condition 1, since raw coal was used in both conditions. Condition 5 was operated under lower space velocity (176 kg/h/m^3 lower than in Condition 1) and lower fresh catalyst concentration (30 W% lower) but using dewaxed-hydrotreated VSOH as solvents. Coal conversion returned to 94.7W% maf coal, the same as obtained in Condition 1.

524°C+ Residuum Conversion

The 524°C+ resid conversion represents the ability of the process to convert high boiling fractions contained in the feedstocks. The resid conversion is an important measure of the process performance or catalytic activity. For the purpose of calculation, all of the maf portion of the feed coal is considered as 524°C+ resid.

As shown in Table 10, the resid conversion values varied between 82.3 and 92.9 W%. From Condition 2 (Period 12) to Condition 3 (Period 17), the resid conversion didn't drop, although make-up catalyst concentration was reduced by 30 W%.

Starting from Condition 4, the space velocity was further reduced to 420 kg/h/m^3 , therefore extinction recycle of 413 °C + VSB was almost achieved. The adjusting of space velocity caused the expected increase in resid conversion by 5.7 W% from Condition 3 to Condition 4.

Although raw coal was used in Conditions 1 and 5, the comparison of 524°C+ resid conversion between two conditions was very difficult, since changes in catalyst concentration, space velocity and solvent quality were made in condition 5 . The effect of solvent dewaxing and hydrotreating on resid conversion was not clear based on the data available.

Distillate Yield (C4-524 °C and IBP-343 °F) and 524°C+ Residuum Yield

During Run ALC-1, C_4 -524°C distillate yield varied between 60.2 and 69.0W% maf coal. As shown in Table 10 and Figure 11, during oil-agglomerated coal feed conditions (Conditions 2 through 4), C_4-524°C distillate yield was increased from 60.2 to 66.1 W% maf coal. The significant increase in distillate yield is believed to be due to the reduction of space velocity from 530 to 420 kg/h/m^3, causing success in extinction recycle of heavy boiling VSB. The highest 524°C+ resid yield was obtained in Condition 2. The inefficiency of the vacuum still may be responsible for high resid yield.

One of the objectives of Run ALC-1 was to maximize the yield of IBP-343°C distillate yield through different means, including coal deashing, solvent dewaxing and hydrotreating and extinction recycle of VSB. As indicated in Table 10, the yield of IBP-343°C light distillate obtained from Condition 4 was higher than from Conditions 1 through 3, because of the reduction in space velocity and the near extinction recycle of 524°C+ resid. It is interesting to note that the highest yield of IBP-343°C light distillate was obtained in Condition 5, possibly demonstrating the benefit of solvent dewaxing and hydrotreating.

Distillate Yield and Selectivity

The *naphtha* yield is defined as the C_4-177°C product fraction and is shown in Figure 12 and Table 10. The naphtha yield increases, in comparing Conditions 1 and 4, from 18.6 to 23.0 W% maf coal together with increases in heavy gas yield. The highest naphtha yield obtained in Condition 4 may be due to the significant reduction in space velocity (to 420 kg/h/m^3).

The *middle distillate* yield is defined as the 177-343°C product fraction and is shown in Table 10 and Figure 12. In comparing the oil-agglomerated coal Conditions 2 and 4, a trend of increase in middle distillate yield was observed. All these results have indicated that the modifications that were made during Conditions 2 through 4 had a positive impact on the process performance.

The *heavy distillate* yield is defined as the 343-524°C product fraction and is shown in Table 10 and Figure 12. The highest heavy distillate yield was obtained from Condition 2.

Selectivity of distillate yields are shown in Table 10 and Figure 13.

Hydrogen Consumption and Light C_1 -C_3 Gas Yield

The hydrogen consumption for the Run ALC-1 is presented in Figure 14 and Table 10. The hydrogen consumption was 7.5W% maf coal in Condition 1. Hydrogen consumption was reduced by about 1.0 W% on switching to oil-agglomerated coal from the raw coal condition. During oil-agglomerated coal conditions, the slightly higher hydrogen consumption in Condition 4 was due to the decrease in space velocity.

The C_1 -C_3 gas yield varied from 9.4 W% to 12.4 W% maf coal. The highest yield of Ç -Ç gas obtained in Condition 4 was due to the reduction in space velocity, causing extensive cracking.

Performance of Dewaxer and Hydrotreater

Prior to the Run ALC-1, HTI prepared about 213 kg DW-HT VSOH. The ASTM D1160 analysis on these dewaxed VSOH and DW-HT VSOH is shown in Table 6. After hydrotreating, the IBP-343°C fraction increased from 22.0 W% to 44.5 W%, implying that significant hydrocracking of dewaxed VSOH occurred during hydrotreating. It seemed that hydrotreating temperature of 371°C was too high.

As a result, in Condition 5, the off-line hydrotreater temperature was reduced to 360°C and dewaxed VSOH flow rate was increased from 500 to 650 g/h. This adjustments appeared to be effective. As shown in Table 21, the hydrogen content in DW-HT VSOH increased from 8.68 W% to 9.74 W% while the IBP-343°C fraction didn't seem to increase significantly. This suggests that the hydrocracking reaction can be suppressed at relatively lower temperature and higher throughput.

First Stage Performance

Coal and 524°C+ Residuum Conversion

As shown in Table 11, coal conversion obtained from first stage reactor (K-1) varied from 94.2-96.9 W%. The highest coal conversion was obtained in Condition 4 due to decrease in space velocity. On an average, first stage coal conversion was 0.5-1.0 W% less than overall coal conversion.

524°C+ resid conversion from the first stage varied from 46.2 to 66.9 W%, about 30W% lower than total resid conversion. It seems that resid conversion depends more on residence time than coal conversion does.

Gas Yield

As shown in Appendix A-E, the yield of C_1 -C_3 gases obtained from first stage varied between 4.0 and 7.2 W% of maf coal, about 50% of the total C_1 -C_3 gas yield (both stages). On an average, the yield of first stage C_4 -C_7 gases was about 42-45 W% of total C_4 -C_7 gas yield.

The yield of COx from the first stage was above 90 W% of total COx production from both stages.

Analyses of Interstage Samples (SOH, PFL and PFC)

The interstage SOH oil contained lower hydrogen but higher sulfur and nitrogen contents than second stage SOH oil (hydrotreated). As shown in Tables 12 & 18, in most conditions, about 50 % of sulfur removal from first stage SOH oil was achieved and nitrogen removal was 75 W% in the on-line hydrotreater. According to the results from previous bench runs performed in HTI, 99 % of heteroatom removal could be achieved if the hydrotreaters were operated efficiently. Compared with second stage SOH oil, the IBP-177°C and 343°C- fractions of the first stage oil were lower, especially for those obtained from oil-agglomerated coal conditions. In addition, an increase in SOH H/C ratio was observed through the second stage.

The interstage PFL was obtained from the laboratory pressure filtration of slurry samples for sample-handling convenience. As shown in Tables 19, the API gravity, IBP, and even elemental analysis for interstage PFL are quite similar as compared to second stage PFL(Table 13, Period 6). However, interstage PFL contains about 10 W% more 524 °C+ resid than second stage PFL, which is consistent with lower resid conversion obtained from interstage. In addition, the cyclohexane and toluene insolubles are higher than second stage PFL.

No significant difference was observed between the properties of interstage PFC and second stage PFC.

PRODUCT QUALITY

Detailed analyses were carried out on liquid products including SOH oil (first stage and 2nd stage), VSOH, VSB, PFL, and PFC produced from work-up Periods. D1160 distillation was performed on daily basis to help fine tune the unit. In addition, for each Period, ash content and quinoline insolubles in PFC were analyzed to control the solid concentration in the recycle stream.

2nd Stage Separator Overhead Product

The SOH oil stream represents the net light distillate (IBP-343°C) from Run ALC-1. While the hydrotreater unit was on-line during the run, a major distillate stream out of the unit was the SOH stream, as the O-1 hot separator overheads, interstage separator overheads, and unit knockouts were being fed directly to the hydrotreater. The properties of SOH oil for the work-up periods are shown in Table 12. The SOH oils had a typical boiling range of 43-401°C. The API gravities were high

(> 33.9), and H/C atomic ratios were also high (> 1.67), especially during oil agglomerated coal conditions. IBP-177°C, the lightest fraction of the SOH-oil, was also higher during oil agglomerated coal conditions than during coal-only conditions. It is observed from the beginning of Run ALC-1 that the hydrotreater was not performing effectively. According to the results from previous runs, sulfur levels below 75 ppm and nitrogen levels below 50 ppm should be obtained after in-line hydrotreating. Starting from Period 3, the sulfur content in second stage was around 1100 ppm as compared to 3000 ppm of first stage SOH oil. This implies that the hydrotreater was not performing efficiently. Unfortunately, a change out of the hydrotreater catalyst during the run was impossible.

Pressure Filter Liquid (PFL) and Pressure Filter Cake (PFC)

From Period 5 of Run ALC-1, the continuous atmospheric still was taken off-line. As a result, the CAS was bypassed and the separator bottoms were collected through a low pressure flash vessel (V-100). The slurry from V-100 was charged to the pressure filter for separation of solids from the heavy liquid product and recycle oil. The liquid oil (PFL) usually contains more than 80 W% 343°C+ including unreacted heavy residuum material. The solids from pressure filtration, PFC, are oil-containing solids, normally extracted with toluene for oil recovery. The oil-free solids were then used for determining the extent of coal conversion based on the solubility of the PFC material in quinoline.

During Run ALC-1, PFL was used as recycle solvent only in Condition 1. Starting from Condition 2, the entire PFL was fractionated into VSOH and VSB. VSOH was partially recycled and extinction recycle of VSB was attempted. As shown in Table 13, The ASTM D1160 distillation indicated that the PFL contained 17 W % IBP-343°C whereas the 524 °C+ resid content was about 29 W%. Elemental analysis showed that the hydrogen content was about 7.6 W% and sulfur content was around 1.03 W%. The amounts of preasphaltenes and asphaltenes in PFL, characterized by solubilities in toluene and cyclohexane respectively, are also shown in Table 13.

Pressure filter cake was analyzed on a daily basis to control the solid concentration. In Condition 1,

the PFC was extracted with toluene prior to recycle which is shown in Table 16. This caused the solid content of toluene-extracted PFC to be high (80-86%). Starting from Condition 2, the PFC was recycled without toluene wash, which caused a decrease in solid content (fluctuated around 60%). The variation of elemental content (C,H,S,N) of PFC was not significant.

Quality of VSOH (Condition 2-5)

From Condition 3 to Condition 5, VSOH was fractionated into $VSOH_1$ and $VSOH_2$ by vacuum distillation. The cut point was set at 343°C. The $VSOH_1$ represents the 343 °C- fraction while $VSOH_2$ consists of 343 °C+ fraction which was being recycled . As shown in Table 13, the API of $VSOH_1$ varied from 10.2 to 16.2, but the API of $VSOH_2$ did not change very much.

The hydrogen contents of $VSOH_1$ and $VSOH_2$ obtained from Condition 5 were higher than those from Conditions 3 and 4, due to the hydrotreating. It is expected that the donatable hydrogen content was enhanced after dewaxing and hydrotrotreating. It is interesting to note that the H/C ratio for $VSOH_1$ obtained during Condition 5 was the highest (1.45) whereas S and N contents were the lowest, indicating that, to a certain extent, dewaxing and hydrotreating had improved the quality of the solvents.

Quality of VSB (Condition 1-5)

The cut-points of the VSB used in each condition were different. The VSB used in recycle stream of Condition 1 was obtained from toluene-extracted oil fractionated at cut point of 390°C. The VSB used in Conditions 2, 3 and 4 was derived from PFL fractioned at cut point of 413 °C whereas VSB generated in Condition 5 was essentially 524°C+.

As shown in Table 14, the analyses of the VSB for Conditions 1 through 4 indicates that API, elemental composition and ASTM D-1160 for all VSB are quite similar, except that higher hydrogen content VSB were produced during oil agglomerated-coal conditions.

Quality of Unrefined Wax (Condition 5)

Dewaxing and hydrotreating of VSOH was performed in Condition 5. The wax sample received from the dewaxing unit contained oil and acetone. After removal of contaminates by chilling in acetone, the unrefined wax yield for each period was measured and appeared to be about 5 W% mf coal. The waxes obtained from Periods 22-25 were characterized by elemental analysis, TGA and GC-MS. As shown in Table 15, the wax produced in Work-up Period 25 contains 13.22% hydrogen and high H/C ratio. 524°C+ resid content, as characterized by TGA, varied from 0.28 to 4.39 W%. For paraffinic compounds, 524°C is about the boiling point of $n\text{-}C_{38}$, suggesting that the waxes generated from coal liquefaction are light waxes with carbon number lower than C_{40}.

This has been confirmed by GC and GC-MS analysis. As shown in Table 15, the carbon number distribution is mainly in the range of C_{18} -C_{36}. These waxes are mixtures of straight chain and

multibranched paraffins according to GC-MS analysis..

Analysis of True Boiling Point (TBP) Fractions (ASTM 2892, modified by HTI)

The SOH and VSOH were fractioned into four true boiling point (TBP) fractions and the individual boiling fractions were combined according to their production rate to produce a single product for each fraction of interest. These fractions were then analyzed for the chemical and physical properties. The TBP fractions were analyzed for each operating conditions. Results are summarized and shown in Tables 22 through 28.

The IBP-177°C fraction of each condition contained about 50 W% paraffins and 40 W% naphthenes; the aromatic and olefinic contents were low. The aromatic content increased with the increase in boiling point.

Phenolic Content and Simulated Distillation of Separator Overhead

Phenolic contents of first stage and second stage SOH were analyzed and the results are presented in Table 29. The water fraction of first stage SOH contained 6-7 times more phenolics than the second stage SOH water. The hydrotreated SOH oil (second stage) contained significant lower phenolics than the first stage, indicating that the use of in-line hydrotreater tends to convert phenolic compounds. If phenolic compounds are considered as valuable products, it may be necessary to separate phenolic compounds from the first stage SOH before hydrotreating.

Simulated distillation of SOH oil fractions was carried out and the results are summarized in Appendix G.

The loss of phenolics and other organics in the SOH water was considered in the calculation of normalized yield. The carbon content of SOH water was analyzed by CONSOL(Appendix H). The results indicated that the carbon loss in SOH water fraction only had a minor influence on the distillate yield (Appendix I).

Characterization of On-Line HTU Catalyst and Solvent Hydrotreating Catalyst

Previous bench run results indicated that >99% heteroatom removal can be achieved. However, it was observed from Period 3 of Run ALC-1 that the sulfur and nitrogen removal from the SOH oil was only 75 W%. The in-line HTU catalyst was characterized after the run and the results are shown in Table 26. A significant reduction in surface area and pore volume was observed, indicating deactivation of the catalyst. It might be possible that during the early part of Condition 1, heavy materials from CAS overhead was fed to HTU causing coking (carbon deposit) of the hydrotreating catalyst.

The surface area and pore volume of solvent hydrotreating catalyst did not change significantly as compared to fresh catalyst.

CONCLUSIONS

From this 25 day, all slurry catalyst bench scale operation, the following major conclusions may be drawn:

1. Using iron (1 W%) and molybdenum (100 ppm) based dispersed catalysts, two-stage liquefaction of Black Thunder coal is a sustainable operation resulting coal conversion of over 95% and C_4-524°C liquid yield of 69 W%maf coal.

2. The oil agglomeration deashing procedure selectively reduced the calcium content of Black Thunder coal. The ash content of raw coal was reduced by 40% from 5.5 to 3.3 % (mf, SO_3-free basis). The oil agglomerated coal was more active. Coal conversion was 3 W% higher than the raw Black Thunder coal. However, due to the use of foreign agglomerating oil and recycling of low boiling materials(inefficiency of the vacuum still), this potential improvement was not observed in the distillation yield.

3. The oil agglomerates were easily re-dispersed in the recycle solvent.

4. Fresh catalyst make-up rate was reduced by 30%, with no apparent detrimental effect in process performance with either raw coal or oil agglomerated coal.

5. Extinction of 343°C material was not achieved. However, as the space velocity was reduced from 670 Kg mf coal/h/m^3 at the beginning of the run to 400 Kg mf coal/h/m^3 as the run progressed, the operation was approaching near extinction of the 413°C materials (0.81 W%).

6. Treatment of recycle solvent was successfully demonstrated, however, steady state operation was not achieved.

RECOMMENDATION FOR FUTURE WORK

Although ALC-01 demonstrated the viability of all slurry catalyst operation, it also identified several areas for potential improvements:

1. Make-up catalyst rate of 7000 ppm of Fe and 70 ppm Mo was demonstrated successfully. However, in order to further reduce catalyst cost, lower catalyst concentrations should be explored.

2. The goal of extincting 343°C+ material was far from being achieved even after significant reduction in feedstock space velocity. This criterion may not be practical for an all slurry catalyst system. Extinction of the 413°C+ was almost accomplished in Condition 4.

3. Feed blending on a 1 to 2 hours basis provided accurate control of the solvent to coal and recycle solid to feed coal ratios. However, in order to maintain a more consistent recycle solvent quality, it is recommended to increase the feed blending time to 4 to 6 hours.

4. Since the beginning of the run, heteroatoms content of distillate products were significantly higher than anticipated, suggesting that the catalyst in the direct-coupled hydrotreater was poisoned. The excessive catalyst deactivation was due to the flooding of the Atmospheric Still with heavy residues and solids. As a result, atmospheric still overheads fed to the direct-coupled hydrotreater contained high boiling materials. For Run ALC-02, a continuous flow single-stage vacuum flash followed by a reduced pressure packed bed still is recommended to provide a sharper cut point for the hydrotreater feed.

5. The generation of recycle solvent for Run ALC-01 was rather complex consisting of hot separator slurry bottoms, toluene extracted oil from the filter cake, and two cuts of vacuum still overheads ($VSOH_1$ and $VSOH_2$). The complexity as well as the time required for each of the recovery operation had slowed down the approach to steady state. The following improvements at the solvent recovery section are recommended.

   - filtration cycle time: a large plate filter, continue with the addition of light distillate to reduce viscosity.
   - vacuum still: a continuous flow vacuum still with inclined half-plate to increase contacting surface.
   - reduced pressure still: a packed column still for providing better separation and sharper cut point of distillates (1^{st} stage overheads and second state distillates) to the hydrotreater.

6. The use of sulfidation agent to activate the catalyst precursors was arbitrarily chosen as 3 W% of mf coal for each stage. This amount has to be further optimized from the cost point of view. Also, the question of whether addition of sulfidation agent is needed for the second stage should be addressed.

Table 1. Analysis of Feed Black Thunder Mine Coal

HTI Designation	HTI 6213
Moisture Content, W%	10.01
Proximate Analysis, W% Dry	
Volatile Matter	43.48
Fixed Carbon	50.52
Mineral Matter	6.00
Ultimate Analysis, W% Dry	
Carbon	70.12
Hydrogen	5.11
Nitrogen	0.99
Sulfur	0.35
Ash	6.19
Oxygen (Diff.)	17.24
H/C Atomic Ratio	0.88

Moisture Content of Oil-Agglomerated Coal (Analyzed by HTI)

OA Coal Bag#	C1	C2	C3	C4	C5	C6	C7	C8	C9	C10
Moisture	5.59	5.90	7.50	5.99	7.49	7.59	7.20	7.89	7.89	9.01

Table 2. Analysis of Agglomerating Oil

HTI Designation	HTI 6460
API Gravity, °	3.6
Elemental Analysis, W%	
Carbon	89.45
Hydrogen	9.16
Sulfur	0.025
Nitrogen	0.103
ASTM D-1160 Distillation, °C	
IBP	392
5 V%	402
10 V%	406
20 V%	413
30 V%	420
40 V%	427
50 V%	434
60 V%	442
70 V%	454
80 V%	460
90V%	481
95 V%	494
98 V%	524
Distribution, W%	
IBP-454°C	66.67
454-524°C	29.23
524°C$^+$	3.72
Loss	0.38

Table 3. Composition of Agglomerate Products From Runs C1-C10 (RUN ALC-1 Feedstocks)

	Bag No.											
	C2	C3	C4	C5	C6	C7	C8	C9	C6+C7	C1	C10	C2-C9 Avg. ±Std. Dev.
Composition, wt % of Whole Sample												
Moisture	5.77	8.10	6.65	7.90	7.64	7.79	8.12	8.67	7.48	5.48	9.32	7.58 ±0.93
Agglom. Oil	24.06	23.40	23.62	23.36	23.46	23.43	23.41	23.34	23.49	23.28	22.95	23.51 ±0.24
Ash, SO_3-free	2.37	2.34	2.47	2.37	2.27	2.33	2.29	2.26	2.35	2.68	2.28	2.34 ±0.07
MAF Coal	67.81	66.15	67.25	66.37	66.63	66.44	66.18	65.73	66.68	68.55	65.45	66.57 ±0.66
MF Coal (MAF Coal + Ash)	70.17	68.50	69.73	68.74	68.90	68.78	68.47	67.99	69.03	71.24	67.73	68.91 ±0.71
Additional Analyses, wt % of Whole Sample, As-Determined												
Carbon	71.61	70.14	71.11	69.88	69.92	70.12	69.76	68.90	69.97	71.57	68.17	70.18 ±0.84
Hydrogen*	6.31	6.61	6.49	6.64	6.58	6.40	6.69	6.48	6.42	6.44	6.55	6.52 ±0.13
Nitrogen	0.82	0.79	0.81	0.80	0.80	0.80	0.81	0.79	0.80	0.82	0.77	0.80 ±0.01
Sulfur	0.71	0.69	0.72	0.67	0.70	0.67	0.69	0.67	0.68	0.63	0.64	0.69 ±0.02
Heating Value, Btu/lb	12812	12570	12686	12592	12647	12624	12553	12435	12578	12819	12216	12640 ±145
Elemental Composition, wt % of MF Coal Portion of Agglomerates (Calculated on Oil-Free, Moisture-Free, Ash-SO_3-Free Basis)												
Ash, SO_3-Free	3.27	3.28	3.46	3.37	3.23	3.27	3.25	3.02	3.37	3.56	3.28	3.27 ±0.13
Carbon	71.36	71.84	71.64	71.22	70.98	71.46	71.26	70.74	70.84	71.29	70.29	71.31 ±0.35
Hydrogen**	5.05	5.31	5.26	5.37	5.30	5.04	5.43	5.09	5.09	5.31	5.15	5.23 ±0.15
Nitrogen	0.96	0.95	0.96	0.96	0.96	0.96	0.98	0.96	0.96	0.96	0.94	0.96 ±0.01
Sulfur	1.01	0.99	1.03	0.97	1.00	0.96	0.99	0.97	0.97	0.88	0.94	0.99 ±0.02
Oxygen by Diff.	18.36	17.62	17.66	18.12	18.53	18.31	18.09	19.21	18.77	18.00	19.41	18.24 ±0.51

* Includes hydrogen from moisture (as-determined basis).

** Corrected to exclude hydrogen from moisture.

Table 4. Major Elemental Analysis of Ash in Oil-Agglomerated Coal*

	Raw Coal	Oil-Aggl. Coal	Raw/Oil-Aggl.
Na_2O	1.75	0.41	4.27
K_2O	0.51	0.53	0.96
CaO	25.78	8.99	2.87
MgO	5.40	1.45	3.72
Fe_2O_3	6.66	8.87	0.75
TiO_2	1.45	2.51	0.58
P_2O_5	1.36	2.13	0.64
SiO_2	37.34	47.84	0.78
Al_2O_3	19.14	26.93	0.71
Total	99.40	99.65	

* Analysis Performed by CONSOL Inc.

Table 5. Analysis of Start-up/Make-up Oil

HTI Designation	Filtered L-814
API Gravity, °	0.40
ASTM D-1160 Distillation, °C	
IBP	309
5 V%	351
10 V%	374
20 V%	394
30 V%	409
40 V%	426
50 V%	437
60 V%	449
70 V%	467
80 V%	507
84 V%	524
Weight Percents	
IBP-343°C	5.47
343-454°C	53.99
454-524°C	22.18
524°C°	18.36
Elemental Analysis, W%	
Carbon	88.96
Hydrogen	8.25
Sulfur	2.22
Nitrogen	0.19
NMR Data	
W% Aromatic Carbon	88.03
W% Cyclic Hydrogen	44.36

Table 6. Properties of Pre-Prepared DW-HT VSOH

Stream	DW-VSOH	DW-HT VSOH	DW-HT VSOH (343°C +, recycled)
API Gravity	3.5	14.6	7.8
IBP,°C	229	60	324
Elemental Analysis			
Carbon			88.10
Hydrogen			8.47
Sulfur			0.76
Nitrogen			0.65
H/C			1.15
ASTM D-1160 Distillation, Composition, W%			
IBP-343°C	22.00	44.53	4.06
343-454°C	59.62	52.16	87.40
454-524°C	14.57	2.27	5.19
524°C+	2.86	0.00	2.76
LOSS	0.95	1.04	0.59

Note: VSOH= 524°C- fraction of PFL;
DW-VSOH= dewaxed VSOH
DW-HT VSOH=dewaxed-hydrotreated VSOH

Table 7. ALC-1 Bench Run (227-94) Detailed Plan

Conditions	1	2	3	4	5
Periods	1-6	7-13	14-17	18-20	21-25
Temperature, °C					
Pretreater	300	300	300	300	300
K-1	440	440	440	440	440
K-2	450	450	450	450	450
In-line Hydrotreater	379	379	379	379	379
Still Cut Point, °C					
V-100 Flasher	329	329	329	329	329
Vacuum Still	N/A	412	412	412	524
Back Pressure, MPa	---------- 17 ----------				
H_2 Flow Rate, Std Liter/h					
K1 Main	---------- 1120 ----------				
K1 Ebb Line	---------- 980 ----------				
K2 Ebb Line	---------- 980 ----------				
Separators	---------- 84 ----------				
Space Velocity					
kg MF coal/h/m^3/reactor	670	560	560	400	480
Coal Flow Rate, g/h					
Raw Coal @ 10% Moisture	1488	---	---	--	1068
OA Coal@8% Moisture and 23.4% Oil	---	1627	1627	1162	---
Recycle Stream Flow Rate, g/h					
Buffer Oils(K1+K2)	107	90	90	64	77
PFL	1678	--	--	--	--
PFC	357	380	380	271	320
VSB	--	941	941	672	457
VSOH(DW-HT 650F+)	--	--	--	--	665
Total Recycle/mf Coal	1.60	1.60	1.60	1.60	1.52
Total Solid/mf Coal	0.20	0.20	0.20	0.20	0.20
Catalyst Addition Rate, g/h					
Fe Catalyst(10 w% Fe)	134	111	79	56	67
Molyvan-A(29w% Mo)	0.46	0.38	0.27	0.19	0.23
TNPS (37 W% S) to K-1	108	90	90	64	78
H_2S to K-2, g/h	40	40	40	40	40

Table 8. Daily Material Balance and Recycle Concentration

Total Material Balance

Conditions	Operating Day 1	2	3	4	5	6	7	Average
1	99.8	96.3	96.1	94.2	96.9	97.0		96.7+/-1.8
2	100.2	99.2	99.2	96.1	100.3	96.4	97.9	98.5+/-1.7
3	91.9	96.3	96.1	95.5				95.0+/-2.1
4	94.7	96.6	92.0					94.4+/-2.3
5	94.5	95.9	98.1	97.1	96.1			96.3+/-1.3

Total Recycle/mf Coal Ratio

Conditions	Operating Day 1	2	3	4	5	6	7	Average
1	2.3	1.6	1.6	1.6	1.6	1.6		1.7+/-0.29
2	1.6	1.6	1.6	1.6	1.9	1.7	1.6	1.7+/-0.13
3	1.7	1.6	1.6	1.6				1.6+/-0.03
4	1.7	1.6	1.6					1.6+/-0.01
5	1.6	1.6	1.6	1.5	1.5			1.6+/-0.07

Recycle Solid /mf Coal Ratio, W%MF Coal

Conditions	Operating Day 1	2	3	4	5	6	7	Average
1	0.0	6.1	5.2	6.5	19.1	22.7		9.9+/-8.9
2	17.5	25.0	18.5	18.3	21.3	20.0	18.7	19.9+/-2.6
3	19.3	19.4	17.1	21.7				19.4+/-1.9
4	21.2	21.6	19.8					20.9+/-1.0
5	20.4	22.6	20.0	15.6	17.9			19.3+/-2.7

Table 9. Composition of Recycle Stream

Conditions:	1	2	3	4	5
Recycle Stream Composition, W% mf coal					
PFL	108	–	--	–	--
Agglomerating Oil	--	35.1	34.3	32.8	–
VSOH(IBP-413°C)	--	11.8	–	--	--
$VSOH_2$(343°C+)	–	--	9.85	16.6	--
VSOH (DW-HT, 343°C)					88.72
VSB(413°C+)	28.4	84.4	84.8	81.7	
VSB(524°C+)					28.6
PFC	26.7*	35.0	34.2	33.0	33.2
Total	163	166	163	164	151
Distribution, W% mf coal					
IBP-260°C	2.8	1.1	0.4	0.1	0.3
260-343°C	16.0	7.3	4.9	3.1	6.1
343-454°C	54.5	70.9	68.6	74.1	83.1
454-524°C	22.5	28.8	27.3	26.2	11.5
Solid Free 524°C+	44.5	39.0	40.3	40.8	31.6
Unconverted Coal	9.0	6.1	7.7	7.0	7.6
SO_3 Free Ash	13.7	13.2	14.0	12.8	10.3
Total	163	166	163	164	151
IOM&Ash/mf coal	0.227	0.193	0.217	0.198	0.179

* Toluene-extracted PFC

Table 10 . Summary of Process Performance

DATE	04/24/96	04/30/96	05/01/96	05/05/96	05/08/96	05/13/96
PERIOD	6	12	13	17	20	25
Coal Type or Bag #'s	Raw Coal	BAG C5 & C6	C6 & C7	C8 & C9	C9 & C1	Raw Coal
CONDITION	1	2	2	3	4	5
NET NORMALIZED YIELDS, W% maf coal						
C1 in Gases	3.26	2.98	4.22	3.41	3.32	2.90 *
C2 in Gases	2.79	3.28	3.39	3.09	3.49	2.90 *
C3 in Gases	3.37	5.01	4.31	4.34	5.59	3.60 *
C1-C3 in Gases	9.42	11.27	11.92	10.84	12.40	9.40 *
C4-C7 in Gases	4.95	5.52	5.19	5.06	6.48	4.17 *
IBP-177 C	13.65	13.40	13.86	14.55	16.53	12.66
177-260 C	11.29	7.71	7.33	7.75	9.70	13.43
260-343 C	23.34	20.13	19.76	23.60	23.88	31.11
343-454 C	14.60	13.77	8.86	12.87	13.24	5.46
454-524 C	1.21	-0.34	1.50	-2.52	-3.75	-1.01
524+ C	4.33	12.34	15.42	10.38	4.56	7.38
Unconverted Coal	5.04	2.16	2.31	2.42	2.51	5.28
Water YLD by Material Balance	13.83	15.23	17.03	16.19	16.20	13.74
COx	5.92	4.28	2.79	4.44	4.27	6.59
NH3	0.73	0.55	0.53	0.62	0.90	1.04
H2S	-0.77	-0.25	-0.27	0.03	-0.09	-1.13
PROCESS PERFORMANCE, W% maf coal						
Hydrogen Consumption	7.5	5.8	6.2	6.2	6.8	8.1
Coal Conversion (SO3 Free)	95.0	97.8	97.7	97.6	97.5	94.7
524+ C Conversion	90.6	85.5	82.3	87.2	92.9	87.5
C4-524 C Distillates	69.0	60.2	56.5	61.3	66.1	65.6
IBP-343 C Distillates	48.3	41.2	41.0	45.8	50.1	57.2
524+C Resid Yield	4.3	12.3	15.4	10.4	4.6	7.4
Desulfurization	121.0	78.7	77.6	84.2	78.6	118.1
Denitrogenation	71.5	45.2	44.7	49.4	70.9	107.7
WAX Product						4.79
PROCESS CONDITIONS						
Space Velocity, kg mf coal/h/m3						
K-1	675	527	558	550	418	466
K-2	675	527	558	550	418	466
Reactor Temperatures, oC						
K-1	442	443	442	443	443	443
K-2	453	453	453	452	452	454
Fresh Catalyst Concentration, metal wppm mf coal						
Mo in Molyvan A	100	101	100	71	68	70
Fe in Fe-based	10000	10000	9870	7120	6810	6980

* Not including small quantities produced in DW-VSOH hydrotreating.

Table 11. First Stage Performance

Conditions	1	2	3	4	5
Coal Conversion					
1st stage	94.2	94.3	96.3	96.9	94.4
Two stage	95.0	97.8	97.6	97.5	94.7
524°C+Resid Conversion					
1st stage	46.2	48.7	66.0	66.9	64.0
Two stage	90.6	85.5	87.2	92.9	87.5
Gas Yield					
C_1-C_3	4.84	4.07	5.96	6.16	6.05
C_4- C_7	2.84	1.71	2.44	2.51	2.45
COx	5.32	2.06	4.08	3.84	6.31

Note: All values are on W% maf coal basis.

Table 12. Properties of Second Stage Separator Overhead

Conditions	t	1	2	2	3	4	5
Periods	5	6	12	13	17	20	25
Gravity, °API	33.9	34.8	41.2	41.8	41.9	40.5	38.3
IBP, °C	45	45	38	43	49	45	90
FBP, °C	392	401	390	399	377	377	380
Elemental Analysis							
Carbon, W%	86.99	87.28	86.06	86.19	86.19	85.53	86.74
Hydrogen, W%	12.48	12.15	12.97	13.02	13.09	12.84	12.79
Sulfur, ppm	1260	1380	1410	1080	1080	1100	1150
Nitrogen, ppm	1190	1950	1130	960	990	960	1070
H/C Ratio	1.72	1.67	1.81	1.81	1.82	1.80	1.77
ASTM D-86 Distillation, Composition, W%							
IBP-177°C	38.0	36.3	48.6	48.7	49.6	39.1	39.3
177-260°C	21.7	22.9	21.5	20.5	20.5	28.6	21.1
260-343°C	26.3	27.6	17.0	16.9	17.0	23.5	26.3
343°C+	13.0	12.2	11.7	11.6	12.0	7.0	12.8
LOSS	1.0	1.0	1.2	2.30	0.9	0.5	0.5

Table 13. Properties of Pressure Filter Liquid and Vacuum Still Overhead

Conditions	1	1	2	2	3			4		5
Periods	5	6	12	13	17			20		25
Streams	PFL	PFL	VSOH	VSOH	VSOH1	VSOH2	VSOH1	VSOH2	VSOH1	VSOH2
Gravity, API	-4.8	-3.7	9.0	10.0	13.8	___	10.2	6.6	16.2	7.2
IBP,oC	218	218	220	225	203	245	227	262	180	318
Elemental Analysis, W%										
Carbon	88.22	88.89	87.83	87.64	86.55	88.53	87.93	89.16	89.03	89.76
Hydrogen	7.31	7.61	9.12	9.20	9.78	9.08	9.24	8.81	10.74	9.30
Sulfur	1.020	0.714	0.187	0.216	0.146	0.166	0.143	0.141	0.115	0.116
Nitrogen	0.950	1.030	0.810	0.800	0.316	0.800	0.318	0.670	0.158	0.205
H/C ratio	1.03	1.03	1.26	1.26	1.36	1.23	1.26	1.19	1.45	1.24
ASTM D-1160 Distillation,W%										
IBP-343oC	13.34	17.43	37.33	53.80	82.55	35.90	54.25	9.71	64.27	10.20
343-454oC	47.09	40.47	61.98	46.00	16.94	63.12	45.65	90.00	33.13	81.60
454-524oC	12.28	12.65	0.40	0.00	0.00	0.69	0.00	0.00	2.60	5.55
524oC+	26.68	28.91	0.00	0.00	0.00	0.00	0.00	0.00	0.00	2.55
Loss	0.63	0.54	0.29	0.20	0.21	0.29	0.10	0.39	0.00	0.10
On 524oC+ Residuum										
Cyclohexane Insolubles,W%		69.34								
Toluene Insoluble,W%		19.77								

Note: VSOH1 represents IBP-343oC fraction of VSOH; VSOH2 represents 343oC+ fraction of VSOH used in recycle.

Table 14. Properties of Vacuum Still Bottoms (VSB)

Conditions	1	1	2	2	3	4	5
Periods	5	6	12	13	17	20	25
Cut Point, °C	399	399	413	413	413	413	524
Gravity °API	N/A	-8.5	-9.6	-7.5	-8.2	-8.6	-15.6
IBP, °C	N/A	388	393	373	373	389	518
Elemental Analysis							
Carbon, W%	85.19	85.97	88.93	89.10	89.20	89.60	89.70
Hydrogen, W%	6.32	6.38	6.92	7.34	7.22	7.01	5.51
Sulfur ,W%	1.81	1.49	0.34	0.35	0.22	0.1	0.258
Nitrogen, W%	0.91	1.16	1.15	1.12	1.18	1.0	1.41
H/C Ratio	0.89	0.89	0.93	0.99	0.97	0.94	0.74
ASTM D-1160 Distillation, Composition, W%							
IBP-343°C		0.0	0.0	0.0	0.0	0.0	0.0
343-454°C	}53.56*	31.84	34.20	41.63	39.20	36.58	0.0
454-524°C		21.53	22.48	19.11	18.29	18.33	2.29
524°C+	46.44	46.30	42.81	38.91	42.07	44.74	97.05
LOSS		0.33	0.51	0.35	0.44	0.35	0.66
On 524°C+ Residuum:							
Cyclohexane Insols, W%				21.13	20.33	16.66	96.10
Toluene Insolubles, W%				5.96	4.40	1.59	17.76

Note: The amount of sample was not enough for D1160 distillation; 524°C+ resid content was obtained from TGA.

Table 15. Properties of Unrefined Wax Product

Periods	22	23	24	25
Elemental Analysis,W%				
Carbon	86.75	84.28	81.47	85.86
Hydrogen	12.22	12.66	13.09	13.22
Sulfur	0.33	0.37	0.87	0.31
Nitrogen	0.32	0.25	0.20	0.21
H/C Ratio	1.69	1.80	1.93	1.85
Product Distribution, W%				
C_{17}-C_{22}				24.8
C_{23} -C_{30}				71.9
C_{31} -C_{36}				3.3
524 °C+ Resid Content, W%	1.49	0.28	4.39	3.31

Table 16. Properties of Pressure Filter Cake

Conditions	1	1	2	2	3	4	5
Periods	5	6	12	13	17	20	25
Toluene Extracted	Yes	Yes	No	No	No	No	No
Elemental Analysis, W%, ash-free basis							
Carbon	70.04	61.85	80.56	83.96	80.65	81.43	84.31
Hydrogen	3.72	3.31	5.66	5.78	5.66	5.89	5.96
Sulfur	14.03	7.52	12.64	10.87	11.05	9.72	4.80
Nitrogen	1.15	0.74	0.85	4.22	0.88	0.72	0.85
H/C	0.63	0.64	0.84	0.83	0.84	0.87	0.85
Composition, W% PFC							
Quinoline Insolubles	93.90	85.21	58.94	55.24	63.45	59.96	53.92
Ash	67.72	62.58	40.31	37.68	40.94	39.44	40.27
Sulfur in QI Ash, W% Ash	10.00	10.00	3.22	3.91	3.41	3.29	10.80
ASTM Ash	60.55	63.44	45.02	40.97	43.06	41.78	40.15
Sulfur in ASTM Ash, W% Ash	7.34	7.48	3.22	3.00	2.02	2.85	9.00

Table 17. Properties of Toluene-Extracted Pressure Filter Cake

Conditions	1	1	2	2	3	4	5
Periods	5	6	12	13	17	20	25
Elemental Analysis, W%, ash-free basis							
Carbon	70.04	61.95	65.54	64.35	65.70	72.85	74.08
Hydrogen	3.72	3.31	2.84	2.78	2.72	3.52	3.98
Sulfur	16.70	11.68	29.25	28.04	26.14	23.09	7.75
Nitrogen	1.15	0.74	0.11	0.17	0.00	0.65	0.85
H/C	0.63	0.64	0.52	0.52	0.5	0.58	0.64
Composition, W% PFC							
Quinoline Insolubles	93.90	85.21	96.23	95.45	99.04	95.62	76.86
Ash	67.72	62.58	61.79	64.68	65.72	61.00	55.89
Sulfur in QI Ash, W% Ash	10.00	10.00	3.40	4.00	3.66	2.73	9.64
ASTM Ash	80.55	63.44	63.76	63.70	63.62	63.10	50.47
Sulfur in ASTM Ash, W% Ash	7.34	7.48	2.66	2.38	2.34	2.67	9.02

Table 18. Properties of First Stage Separator Overhead

Conditions	**1**	**2**	**3**	**4**	**5**
Periods	6	13	17	20	25
Gravity, °API	28.4	31.8	31.6	28.1	29.1
IBP, °C	54	54	62	58	55
FBP, °C	404	411	372	383	380
Elemental Analysis,W%					
Carbon	84.39	83.03	84.19	84.24	84.81
Hydrogen	11.21	11.54	11.74	11.29	11.43
Sulfur	0.209	0.174	0.166	0.149	0.287
Nitrogen	0.428	0.450	0.431	0.423	0.311
H/C Ratio	1.59	1.67	1.67	1.61	1.62
ASTM D-86 Distillation, Composition, W%					
IBP-177°C	37.3	42.8	41.9	34.6	35.1
177-260°C	26.2	22.8	23.7	25.8	19.9
260-343°C	21.4	17.8	17.4	22.7	16.2
343°C+	13.8	15.7	16.3	16.2	28.1
LOSS	1.3	0.9	0.7	0.7	0.7

Table 19. Properties of First Stage Sample-Pressure Filter Liquid

Conditions	**1**	**2**	**3**	**4**	**5**
Periods	6	13	17	20	25
Gravity °API	-4.2	-2.0	-3.4	-10.5	-3.2
IBP, °C	245	246	255	234	257
Elemental Analysis, W%					
Carbon	88.32	88.11	88.65	88.86	89.41
Hydrogen	7.56	7.98	7.77	7.65	7.59
Sulfur	0.65	0.36	0.42	0.38	0.19
Nitrogen	1.16	1.11	1.03	1.05	0.92
H/C Ratio	1.03	1.09	1.05	1.03	1.02
ASTM D-1160 Distillation, Composition, W%					
IBP-343°C	10.71	11.27	8.15	7.22	9.07
343-454°C	34.64	40.01	39.82	43.43	44.46
454-524°C	12.86	15.27	16.59	14.14	11.25
524°C+	41.61	32.36	34.54	34.70	34.66
LOSS	0.18	1.09	0.90	0.51	0.56
On 524 °C+ Residuum:					
Cyclohexane Insols, W%	79.81	79.66	78.31	79.15	67.20
Toluene Insolubles, W%	22.41	28.41	21.34	22.54	22.97

Table 20. Properties of First Stage Pressure Filter Cake

Conditions	1	2	3	4	5
Periods	6	13	17	20	25
Elemental Analysis, W%, ash-free basis					
Carbon	81.74	81.16	87.89	83.05	91.24
Hydrogen	5.90	6.00	6.33	5.61	6.73
Sulfur	6.82	9.11	5.96	6.48	3.84
Nitrogen	1.12	1.12	1.10	0.87	0.91
H/C	0.87	0.89	0.86	0.81	0.88
Composition, W% PFC					
Quinoline Insolubles	58.30	58.24	61.48	61.74	56.32
Ash	44.56	41.14	45.60	44.50	40.98
Sulfur in QI Ash, W% Ash	10.50	5.14	4.90	4.40	9.10
ASTM Ash	44.58	41.82	45.67	44.79	40.55
Sulfur in ASTM Ash, W% Ash	9.09	4.04	5.13	3.62	7.76
TGA 524oC Resid, W%	71.56	72.88	70.81	73.34	64.95

Table 21. Properties of Dewaxed and Hydrotreated Solvent

(Condition 5, Period 25)

Stream	VSOH	DW-VSOH	DW-HT VSOH	
			Whole	343°C+
API Gravity	5.0	3.7	9.9	7.2
IBP, °C	230	253	180	318
Elemental Analysis, W%				
Carbon	89.51	88.95	89.54	89.76
Hydrogen	8.68	8.34	9.74	9.30
Sulfur	0.15	0.17	0.12	0.12
Nitrogen	0.53	0.53	0.19	0.21
H/C Ratio	1.16	1.13	1.31	1.24
ASTM D-1160 Distillation, Composition, W%				
IBP-343°C	20.48	21.81	26.64	10.20
343-454°C	71.52	72.38	66.86	81.60
454-524°C	5.30	3.61	4.65	5.55
524°C+	2.60	1.91	1.77	2.55
LOSS	0.10	0.29	0.08	0.10

Table 22. Analysis of TBP Fractions: Condition 1, Period 5

TBP Distillation

IBP,°C	47
Distribution	W%
IBP-177°C	39.0
177-260°C	21.1
260-343°C	29.0
343°C+	10.5
LOSS	0.4

	IBP-177°C	177-260°C	260-343°C	343+°C
API Gravity	54.7	31.0	19.5	11.5
Elemental Analysis, W%				
Carbon	85.19	87.96	88.90	89.77
Hydrogen	14.46	12.46	11.13	9.92
Sulfur, ppm	1090	1110	1150	1370
Nitrogen, ppm	1020	1310	1160	1400
Bromine No. [g/100g]	1.05	2.06	1.92	
Aniline Point, [°C]	51	32	24	
Pour Point, [°C]	-62	-62	-50	
Flash Point, [°C]	<-18	70	138	
PONA[V%]				
Paraffins	51.74	15.79		
Olefins	0.50	1.40		
Naphthenics	40.99	42.55		
Aromatics	6.77	40.26	61.74	

Table 23. Analysis of TBP Fractions: Condition 1, Period 6

TBP Distillation,

IBP, °C	42

Distribution	W%
IBP-177°C	36.6
177-260°C	25.0
260-343°C	31.0
343°C+	7.3
LOSS	0.1

	IBP-177°C	177-260°C	260-343°C	343+°C
API Gravity	55.1	28.7	18.4	8.6
Elemental Analysis, W%				
Carbon	84.74	88.94	89.25	90.14
Hydrogen	14.33	11.94	10.74	9.46
Sulfur, ppm	1100	1140	1340	1740
Nitrogen, ppm	1220	1400	1400	1600
Bromine No. [g/100g]	3.95	14.02	5.61	
Aniline Point, [°C]	51	19	17	
Pour Point, [°C]	-62	-62	-52	
Flash Point, [°C]	<-17	68	138	
PONA[V%]				
Paraffins	50.58	14.46		
Olefins	0.60	1.50		
Naphthenics	41.81	38.04		
Aromatics	7.01	46.0	65.6	

Table 24. Analysis of TBP Fractions: Condition 2, Period 12

TBP Distillation,

IBP, °C	49
Distribution	W%
IBP-177°C	19.6
177-260°C	19.9
260-343°C	26.6
343°C+	33.6
LOSS	0.3

	IBP-177°C	177-260°C	260-343°C	343+°C
API Gravity	55.6	28.0	14.9	7.4
Elemental Analysis, W%				
Carbon	85.16	84.11	88.23	89.23
Hydrogen	14.72	11.31	10.34	9.21
Sulfur, ppm	1090	1210	1570	1780
Nitrogen, ppm	990	1510	1710	1660
Bromine No. [g/100g]	0.53	12.6	21.07	
Aniline Point, [°C]	54	24	13	
Pour Point, [°C]	-62	-46	-10	
Flash Point, [°C]	<-17	51	134	
PONA[V%]				
Paraffins	52.54	15.10		
Olefins	0.70	N/A		
Naphthenics	41.83	35.56		
Aromatics	4.93	49.34	71.1	

Table 25. Analysis of TBP Fractions: Condition 2, Period 13

TBP Distillation

IBP, °C	39
Distribution	W%
IBP-177°C	28.6
177-260°C	18.0
260-343°C	24.0
343°C+	29.0
LOSS	0.4

	IBP-177°C	177-260°C	260-343°C	343+°C
API Gravity	55.9	28.6	15.6	7.8
Elemental Analysis, W%				
Carbon	84.84	87.02	88.17	89.11
Hydrogen	14.57	11.81	10.33	9.29
Sulfur, ppm	1090	1230	1510	1770
Nitrogen, ppm	990	1590	1580	1670
Bromine No. [g/100g]	0.53	18.53	20.76	
Aniline Point, [°C]	52	24	13	
Pour Point, [°C]	-62	-51	-8	
Flash Point, [°C]	<-17	47	138	
PONA[V%]				
Paraffins	50.55	14.35		
Olefins	0.60	N/A		
Naphthenics	43.98	36.77		
Aromatics	4.87	48.88	67.7	

Table 26. Analysis of TBP Fractions: Condition 3, Period 17

TBP Distillation

IBP, °C	42

Distribution	W%
IBP-177°C	25.1
177-260°C	18.9
260-343°C	24.8
343°C+	30.9
LOSS	0.3

	IBP-177°C	177-260°C	260-343°C	343+°C
API Gravity	57.2	26.1	14.6	7.8
Elemental Analysis, W%				
Carbon	84.94	86.24	87.88	89.16
Hydrogen	14.70	11.27	10.04	9.21
Sulfur, ppm	1090	1200	1420	1510
Nitrogen, ppm	996	1620	1560	1650
Bromine No. [g/100g]	1.07	33.81	18.56	
Aniline Point, [°C]	53	19	9	
Pour Point, [°C]	-62	-60	-12	
Flash Point, [°C]	<-17	69	138	
PONA[V%]				
Paraffins	52.25	13.57		
Olefins	0.60	N/A		
Naphthenics	41.92	32.80		
Aromatics	5.23	53.63	70.6	

Table 27. Analysis of TBP Fractions: Condition 4, Period 20

TBP Distillation,

IBP, °C	41

Distribution	W%
IBP-177°C	27.4
177-260°C	20.3
260-343°C	29.4
343°C+	22.2
LOSS	0.7

	IBP-177°C	177-260°C	260-343°C	343+°C
API Gravity	55.8	28.4	15.8	6.9
Elemental Analysis, W%				
Carbon	85.0	87.19	88.44	89.47
Hydrogen	14.60	11.80	10.41	9.26
Sulfur, ppm	1100	1160	1280	1440
Nitrogen, ppm	972	1610	1600	1670
Bromine No. [g/100g]	0.53	19.86	19.55	
Aniline Point, [°C]	51	24	9	
Pour Point, [°C]	-62	-58	-12	
Flash Point, [°C]	<-17	64	129	
PONA[V%]				
Paraffins	47.61	13.48		
Olefins	0.60	N/A		
Naphthenics	46.44	37.95		
Aromatics	5.35	48.57	69.8	

Table 28. Analysis of TBP Fractions: Condition 5, Period 25

TBP Distillation,

IBP, °C	43
Distribution	W%
IBP-177°C	26.2
177-260°C	18.2
260-343°C	35.0
343°C+	20.6
LOSS	0.0

	IBP-177°C	177-260°C	260-343°C	343+°C
API Gravity	56.3	30.4	17.6	6.7
Elemental Analysis, W%				
Carbon	84.91	88.09	89.22	90.54
Hydrogen	14.63	12.24	11.11	9.48
Sulfur, ppm	1090	1100	1110	1150
Nitrogen, ppm	927	1190	1340	1440
Bromine No. [g/100g]	0.27	1.60	1.68	
Aniline Point, [°C]	52	26	18	
Pour Point, [°C]	-62	-62	-52	
Flash Point, [°C]	<-17	66	131	
PONA[V%]				
Paraffins	51.16	8.22		
Olefins	0.60	1.4		
Naphthenics	42.48	48.98		
Aromatics	5.76	41.4	55.0	

Table 29. Phenolic Content of Separator Overhead

Conditions	1	1	2	2	3	4	5
Periods	5	6	12	13	17	20	25
			Phenolic Content, ppm				
Water Fraction							
First stage	N/A*	2920	N/A*	3350	3180	3580	4660
Second stage	490	1480	770	470	610	360	120
Oil Fraction							
First stage	N/A*	18600	N/A*	23700	23200	22500	22100
Second stage	440	760	530	480	900	300	250

N/A: Samples were not available

Table 30. Characterization of Hydrotreater Catalyst
(Criterion C-411)

Catalyst	Fresh	In-line HTU (Spent)	Solvent Hydrotreating (Spent)
Surface Property			
BET Surface Area, m^2/g	152	115	151
Pore Volume, ml/g	0.387	0.153	0.379
Elemental Analysis, W%			
Carbon	0.17	10.63	0.45
Hydrogen	0.47	0.93	0.44
Sulfur	0.10	6.49	0.10
Nitrogen	0.04	0.24	0.10
Metals, W% Catalyst			
Ni	2.22	1.63	2.18
Mo	13.01	9.61	12.84

Table 31. Comparison of Run ALC-1 and CMSL-9

Run No	ALC-1	CMSL-9
Conditions	1	6
Period	6	29
Coal	Black Thunder	Black Thunder
Space Velocity, kg/hr/m³ mf coal, each stage	683	666
Reactor Temperature, °C		
K-1	442	440
K-2	453	449
Recycle Streams, W% mf coal		
IBP-343 °C	18.8	35.9
343-454 °C	54.5	66.0
454-524 °C	22.5	3.3
Solid Free 524 °C+	44.5	16.2
Unconverted Coal	9.0	5.3
SO_3 Free Ash	13.7	10.8
Recycle/MF Coal	1.63	1.38
Catalyst Concentration, ppm mf coal		
Mo	100	100
Fe	10000(gel)	10000(powder)
Net Normalized Yield, W% maf coal		
C_1-C_3 in Gases	9.42	9.23
C_4-C_7 in Gases	4.95	4.66
IBP-177°C	13.65	7.37
177-260°C	11.29	15.64
260-343°C	23.34	14.92
343-454°C	14.60	12.44
454-524°C	1.21	4.01
524°C+	4.33	7.35
Unconverted Coal	5.04	4.34
H_2O	13.83	14.96
COx	5.92	4.38
NH_3	0.73	0.86
H_2S	-0.77	-0.15
Process Performance, W% maf Coal		
Hydrogen Consumption	7.5	5.8
Coal Conversion	95.0	96.1
524°C+ Conversion	90.6	87.6
C4-524°C Distillate	69.0	62.6
524°C+ Resid Yield	4.3	7.4

Table 32

RUN ALC-1 OPERATIONS-STATUS-CLASSIFICATION CHRONOLOGY

Operations Classification I.D.	Start Time	Start Date	End Time	End Date	Duration, Hours
S/U	1600	4/15/96	1700	4/16	25
S/D	1700	4/16	1800	4/16	1
DT	1800	4/16	1400	4/17	20
S/U	1400	4/17	0500	4/19	39
1-25	0500	4/19	0400	5/14	599
S/D	0400	5/14	1000	5/15	30

Table 33

ON-LINE TIME AND DOWN TIME FOR RUN ALC-1

Time of Initial S/U & Final S/D, Hours	Time of Intermediate S/Ds & S/Us, Hours (No. of Int. S/D-S/U) Hours	Total On-Line Time, Hours	Down-Time, Hours	On-Line Efficiency, %
55	40	694	20	97.2

Definitions:

Startup: (S/U) Time between gas flow initiation and feedstock cut-in during which unit temperatures and/or pressures are being increased.

Shutdown: (S/D) Time between feedstock cut-out and liquid flow termination during which unit temperatures and/or pressures are being decreased.

Run-Periods: Time during which the unit is at run conditions and the operations is identified with a Period number.

On-Line Time: The sum of S/Us, S/Ds, and Run-Periods.

Down-Time: (DT) The time during which gases and liquids are not being charged to the unit. This is the same as the time between an intermediate shutdown and startup.

On-Line Efficiency=On-Line Time / (On-line Time + Down-Time)

Figure 1. Simplified Schematic of HTI's Bench Unit

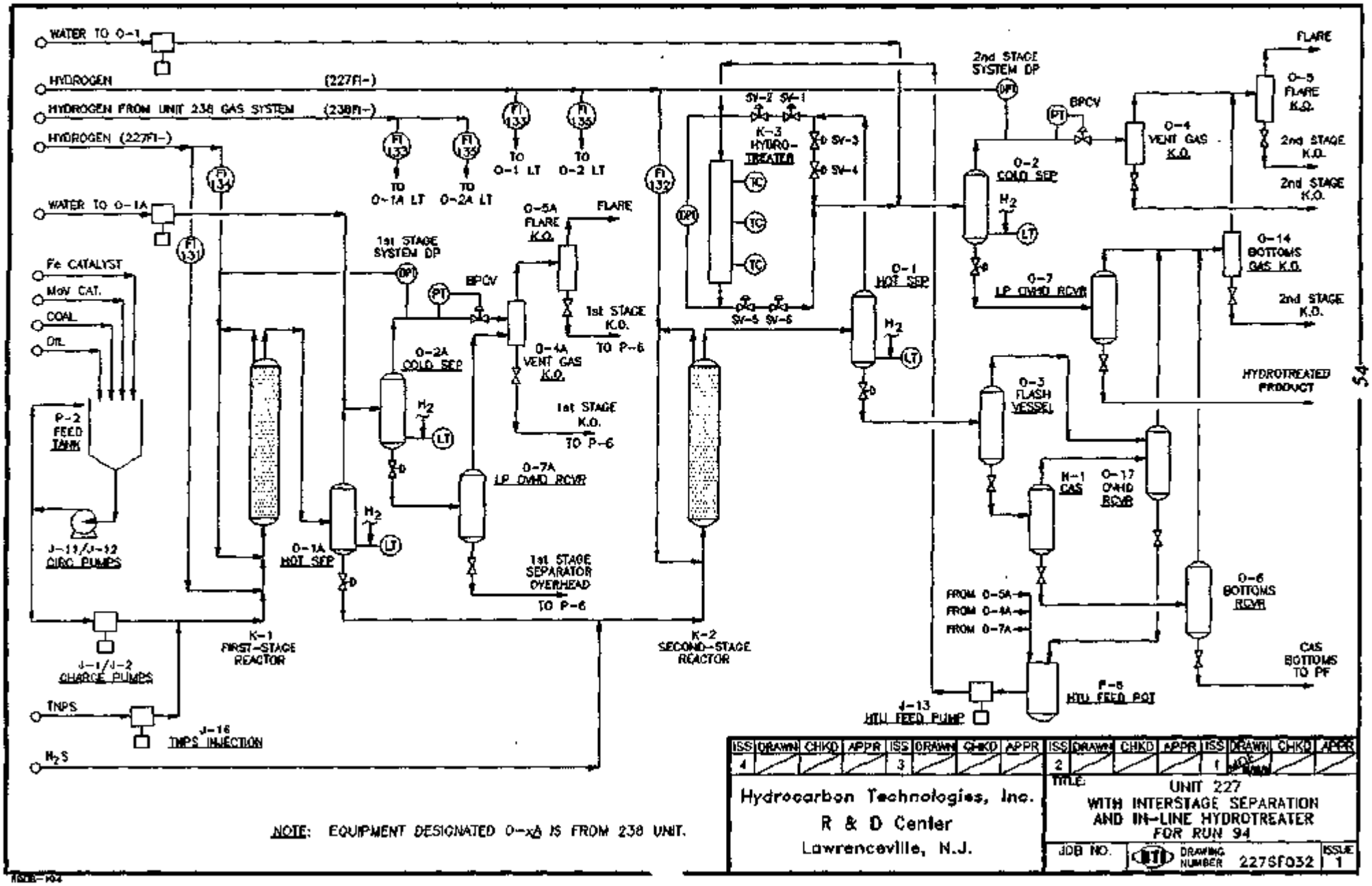

Figure 2

Downstream Product Flow Scheme
Condition 1 (Periods 1-7)

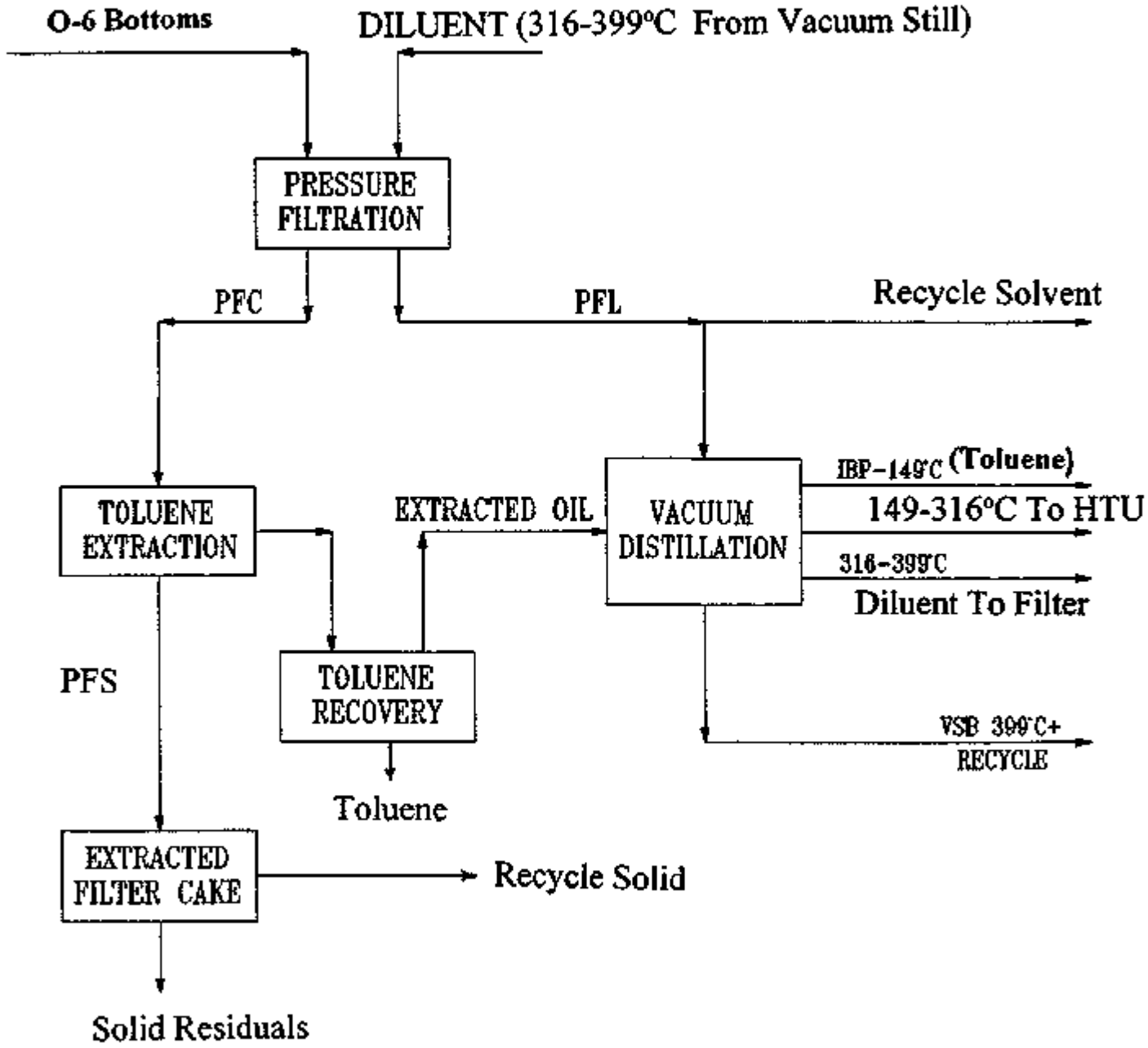

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Figure 3

Downstream Product Flow Scheme
Condition 2 (Periods 8-13)

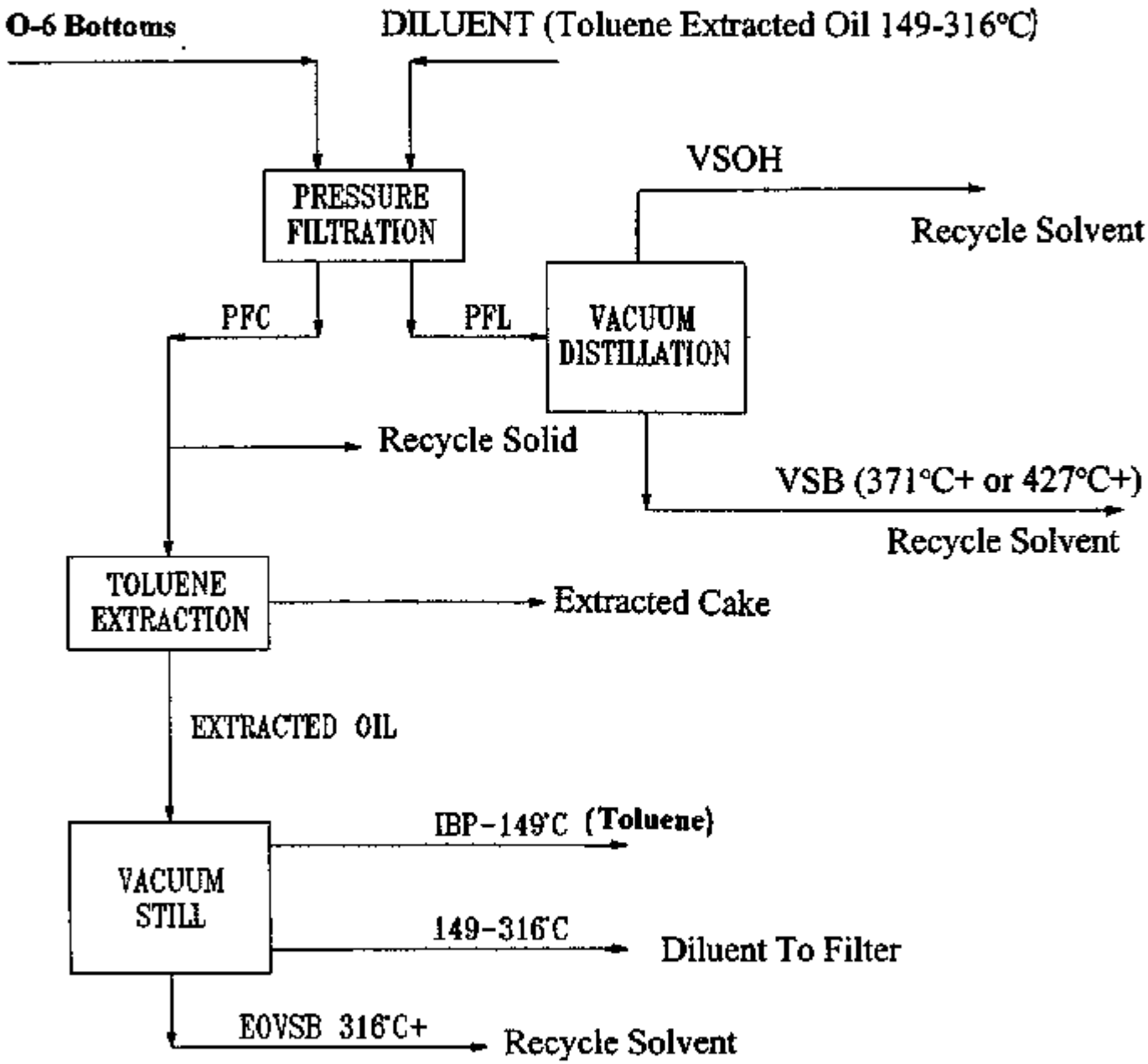

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Figure 4

Downstream Product Flow Scheme
Conditions 3&4 (Periods 14-20)

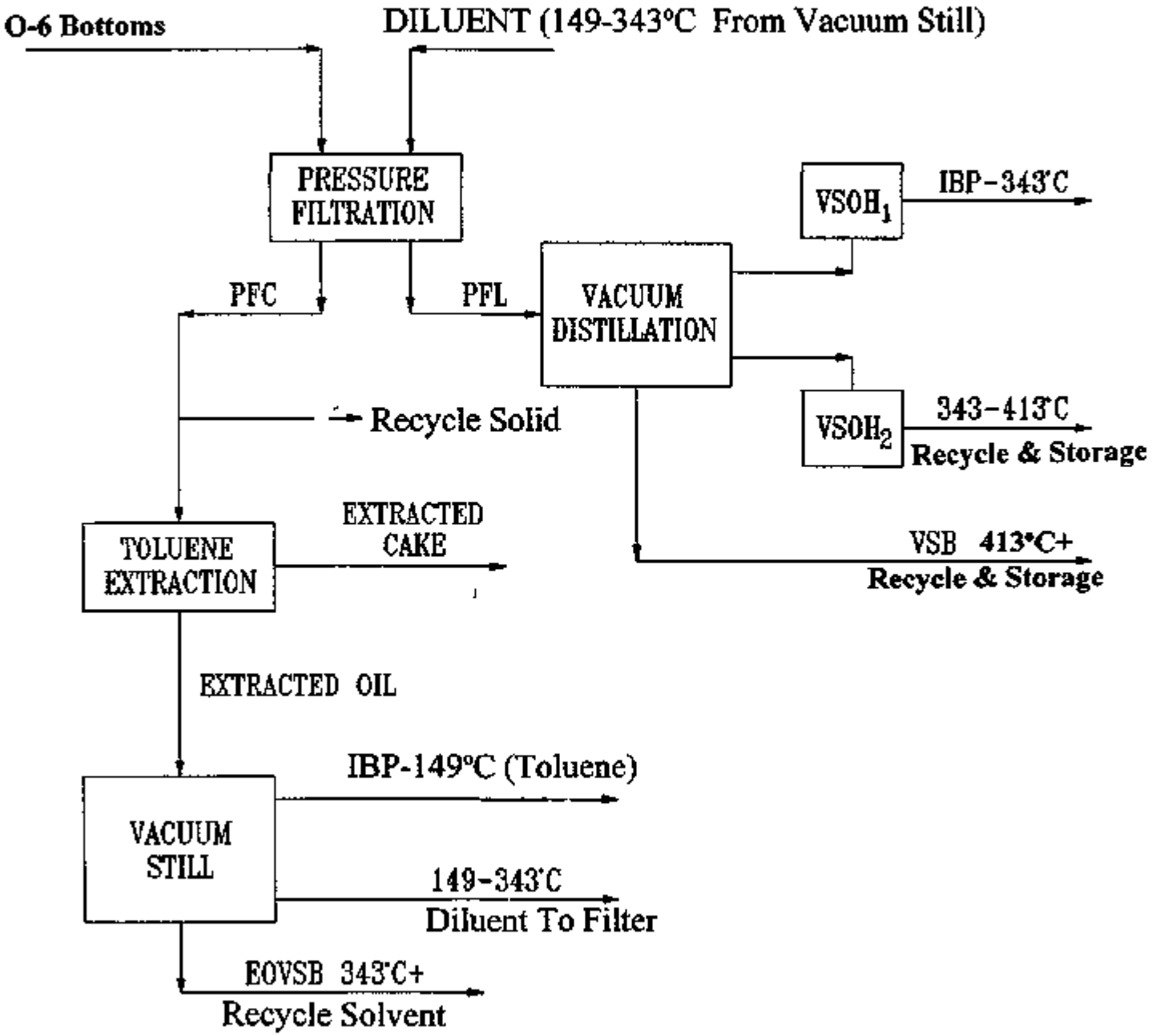

Figure 5

Downstream Product Flow Scheme
Condition 5 (Periods 21-25)

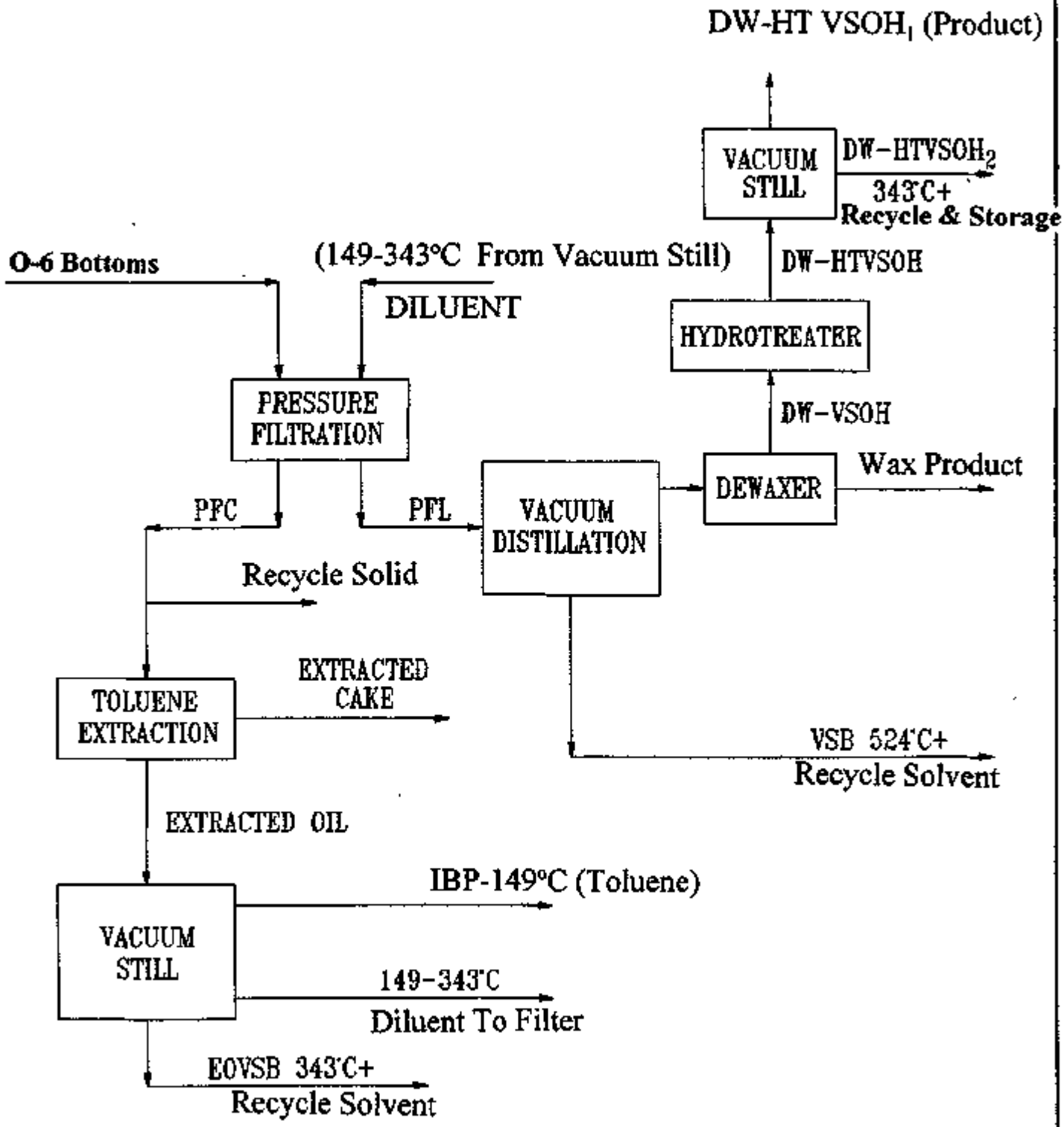

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Figure 6. Simplified Schematic of HTI's Ketone Dewaxing Unit

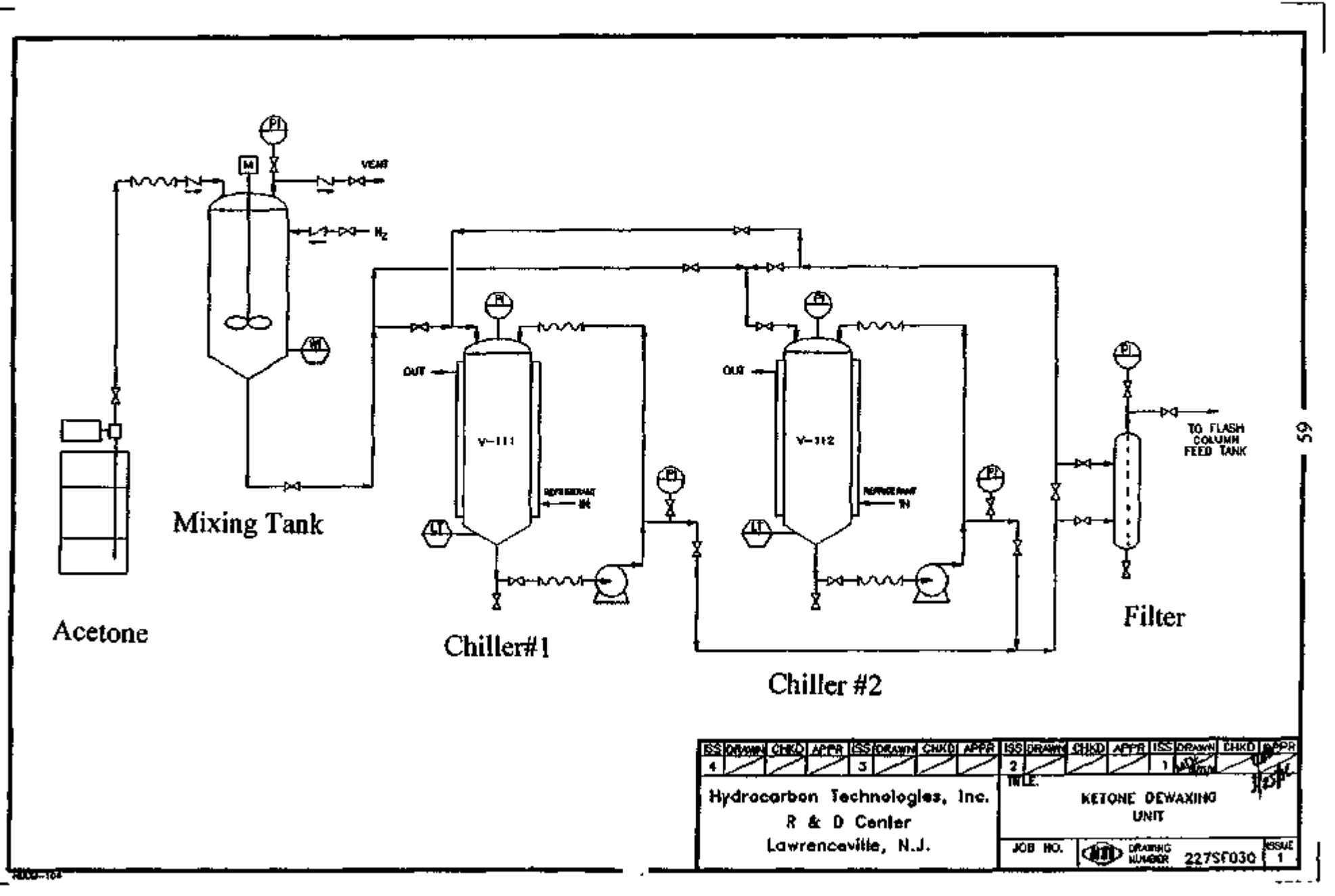


Figure 7

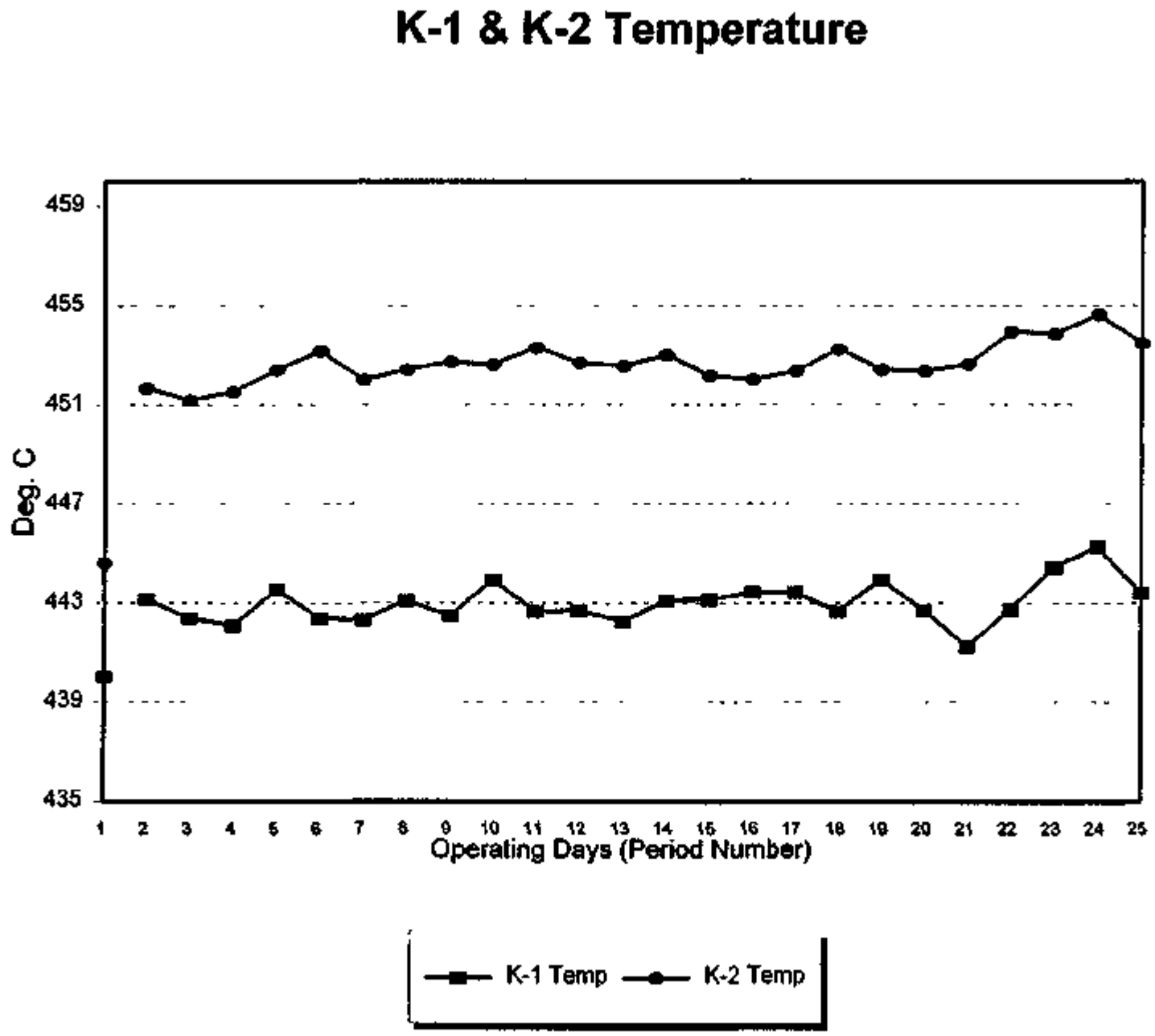

K-1 & K-2 Temperature
459
455
451
447
443
439
435
Deg. C
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25
Operating Days (Period Number)
K-1 Temp
K-2 Temp

Figure 8

O-1A & O-1 Temperature

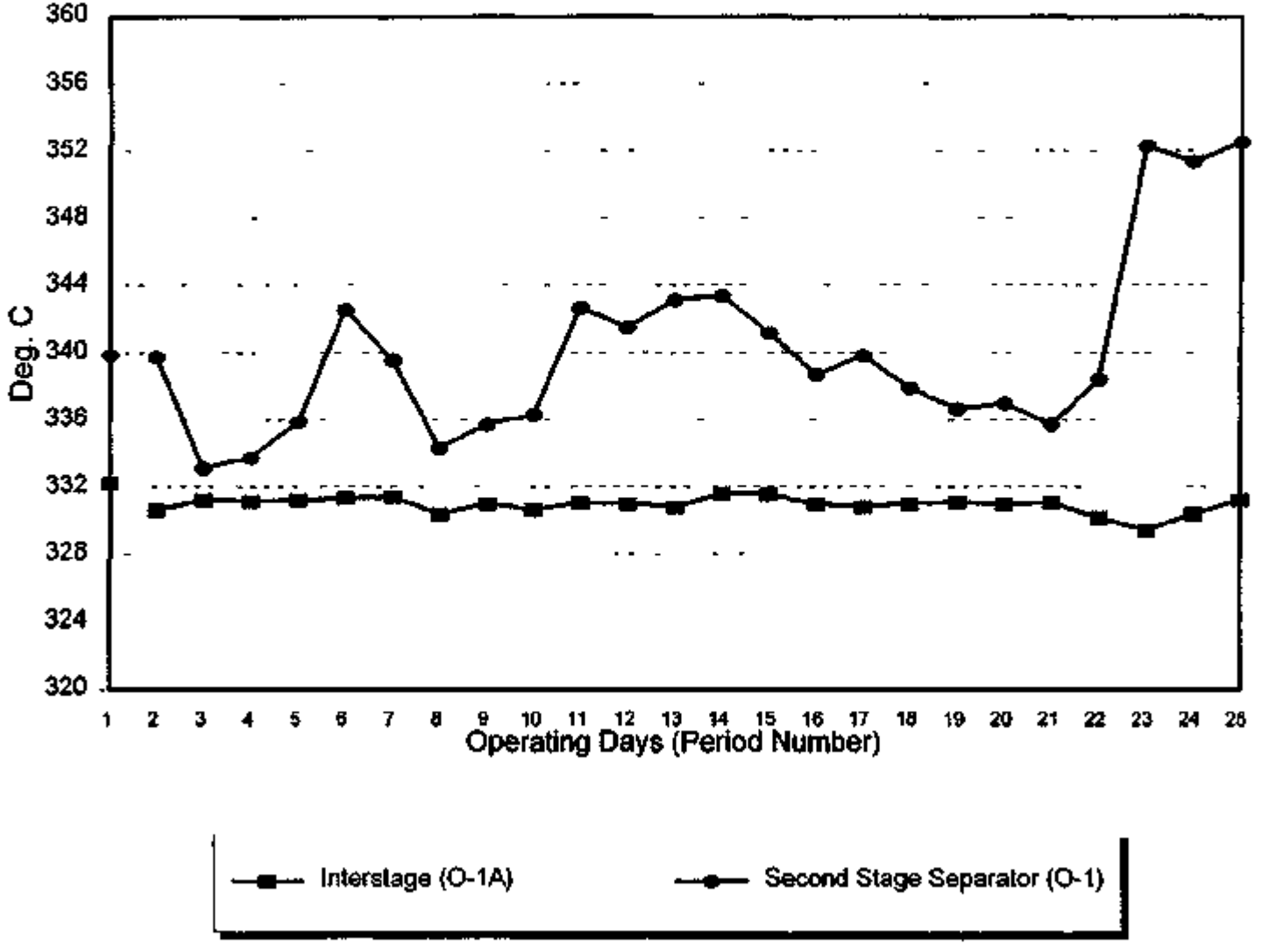


Figure 9

Space Velocity

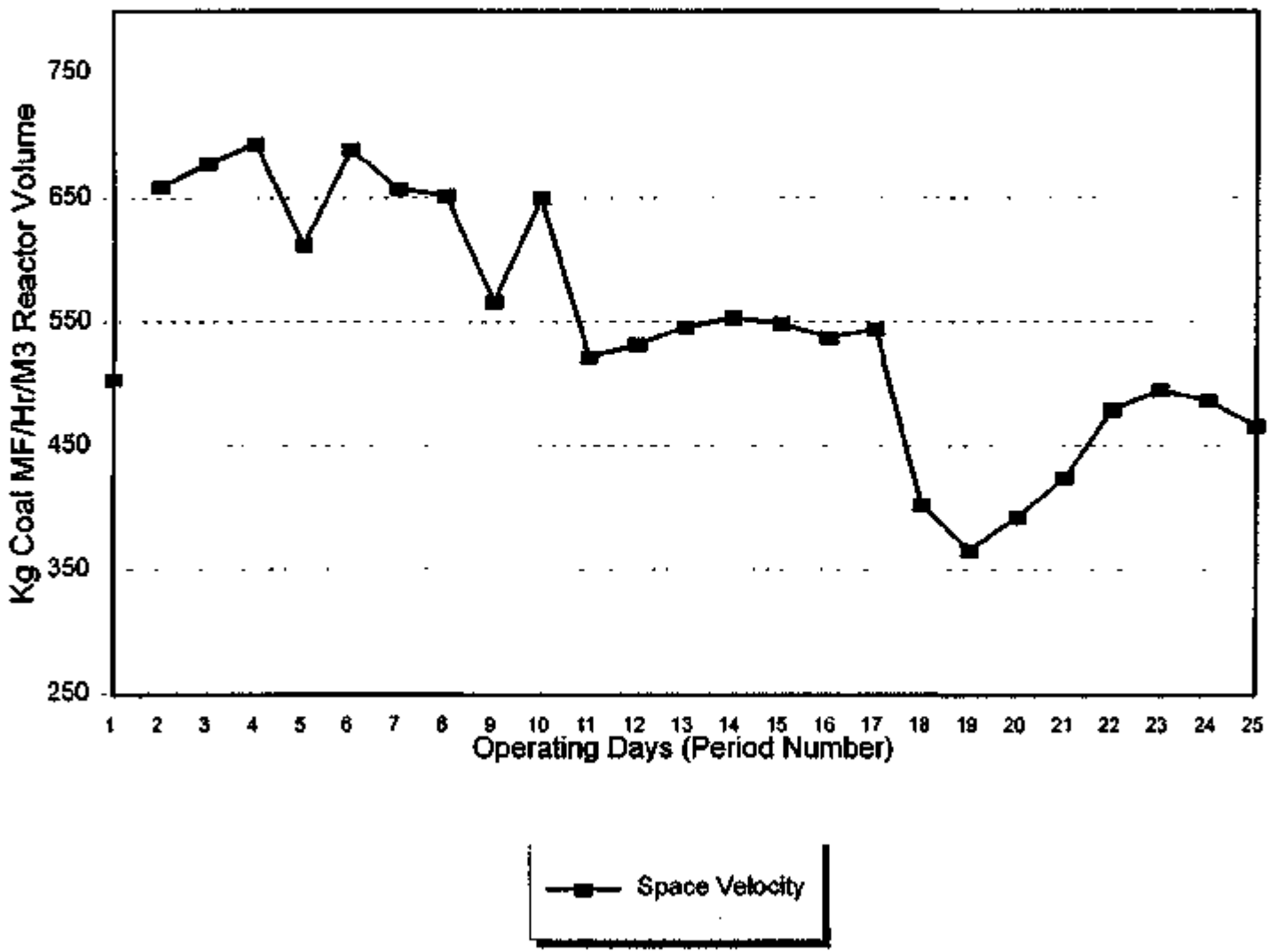


Figure 10

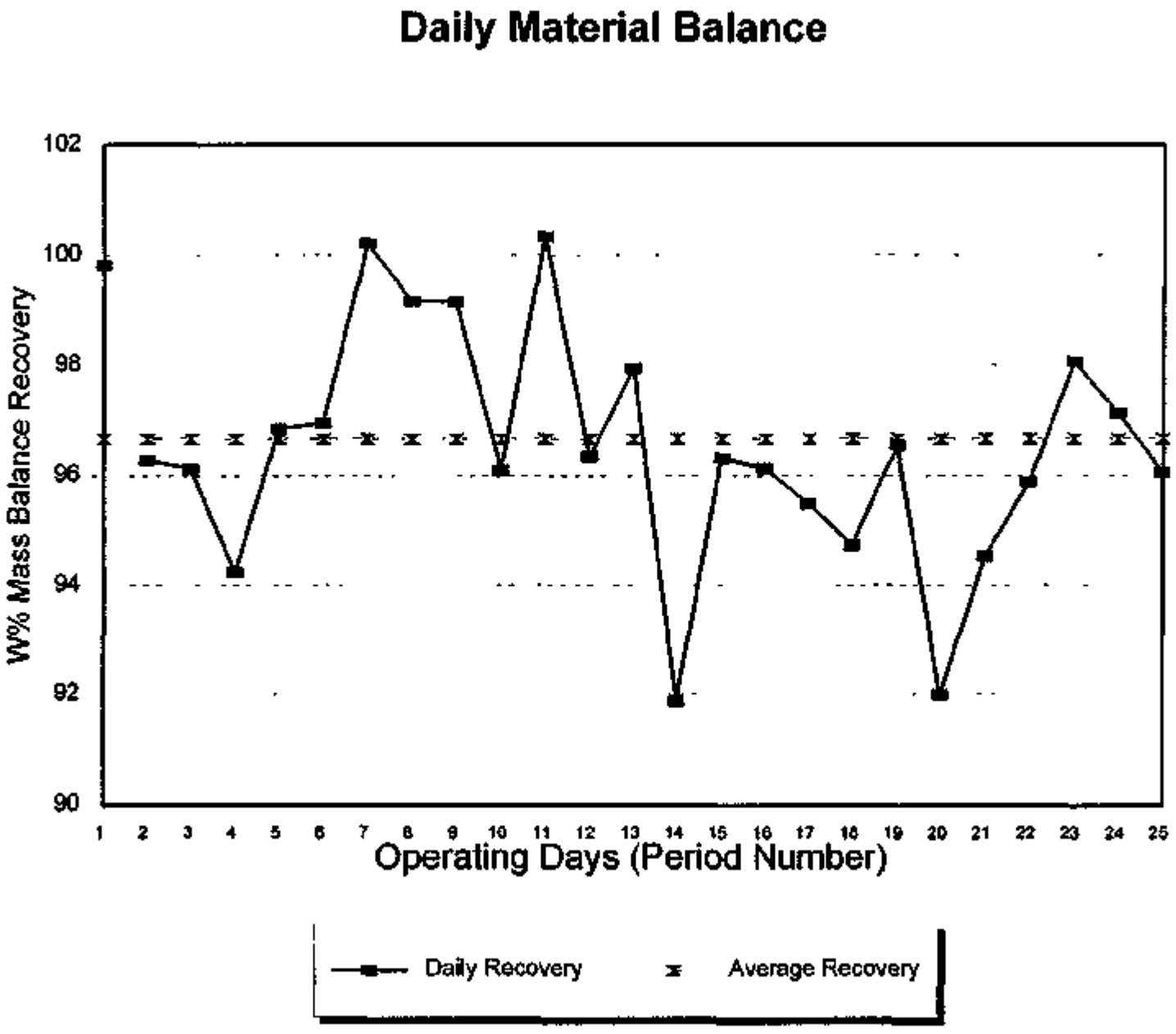

Daily Material Balance
W% Mass Balance Recovery
102
100
98
96
94
92
90
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25
Operating Days (Period Number)
Daily Recovery
Average Recovery

Figure 11

C4-524 C Distillate Yield & 524 C+ Yield

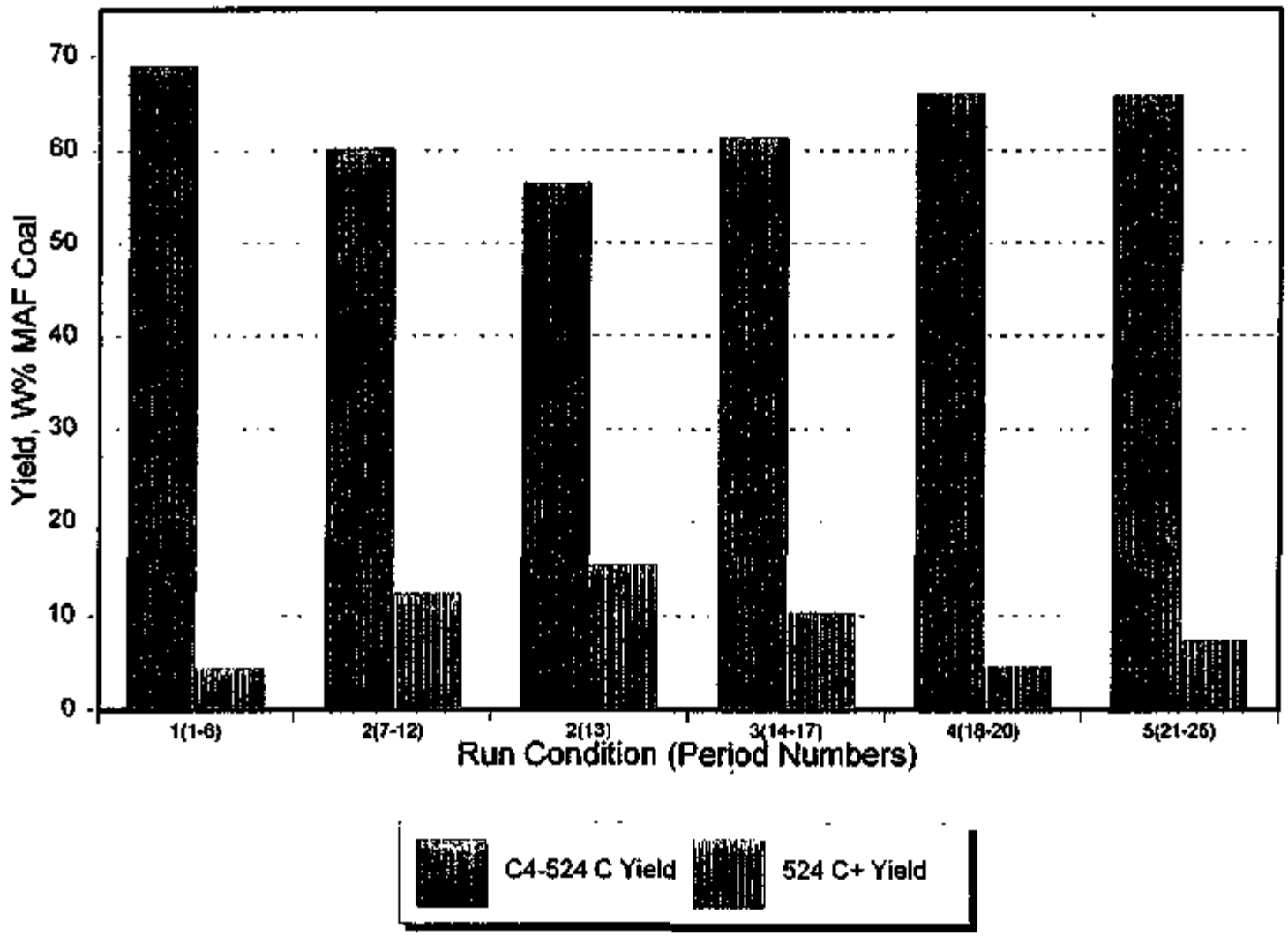


Figure 12

Distillate Fraction Yields

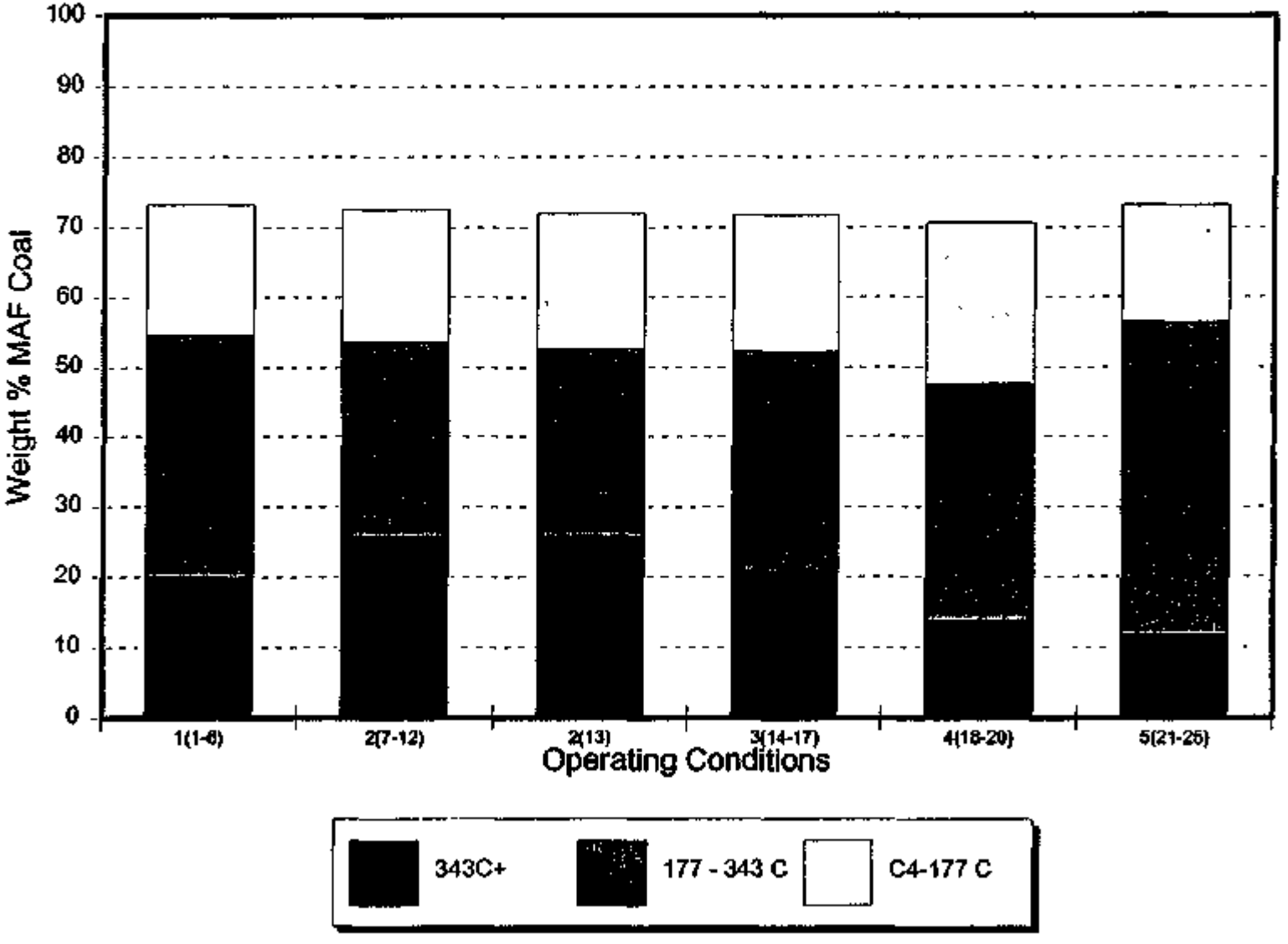


Figure 13

Distillate Fraction Selectivity

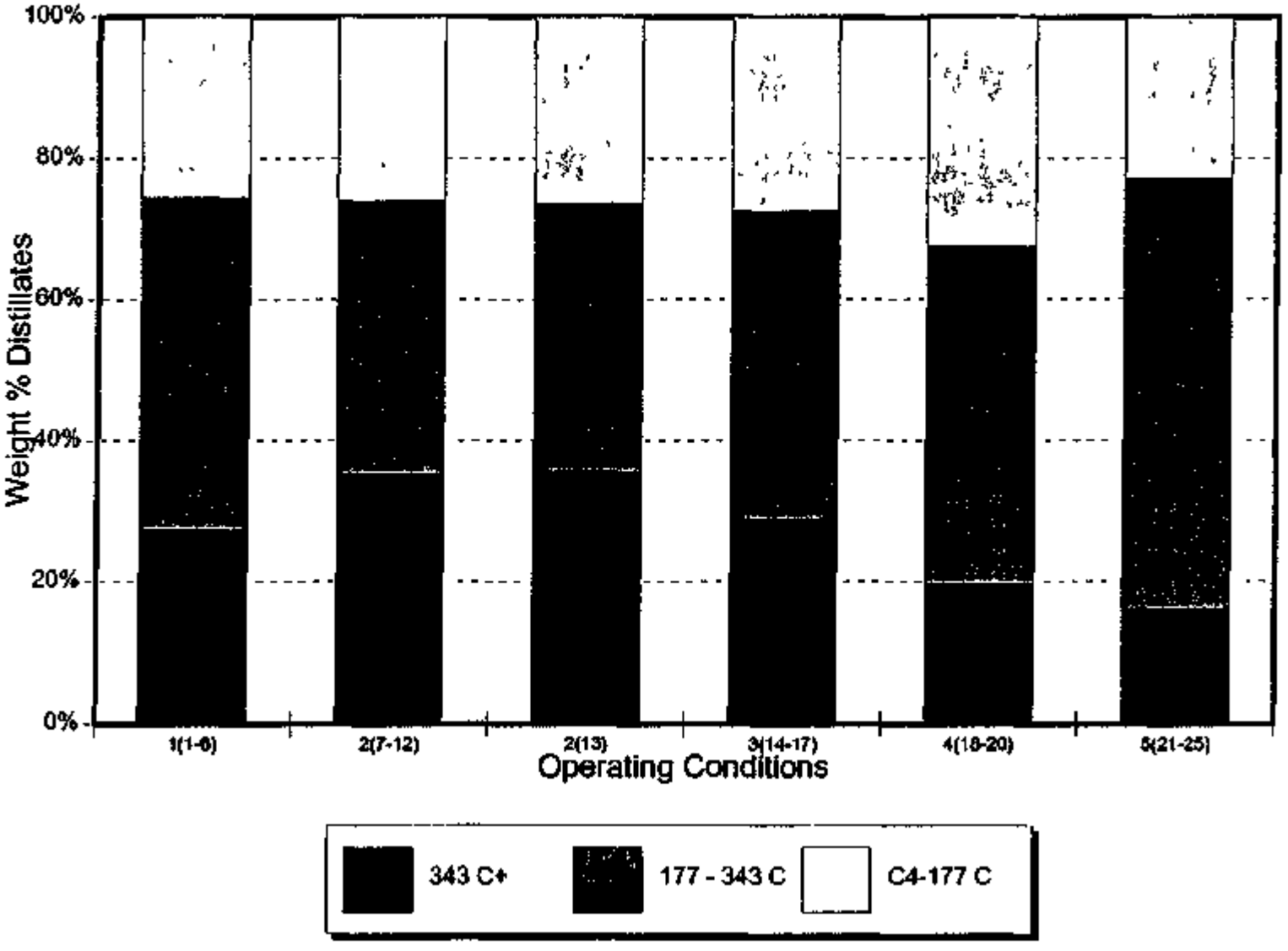


Figure 14

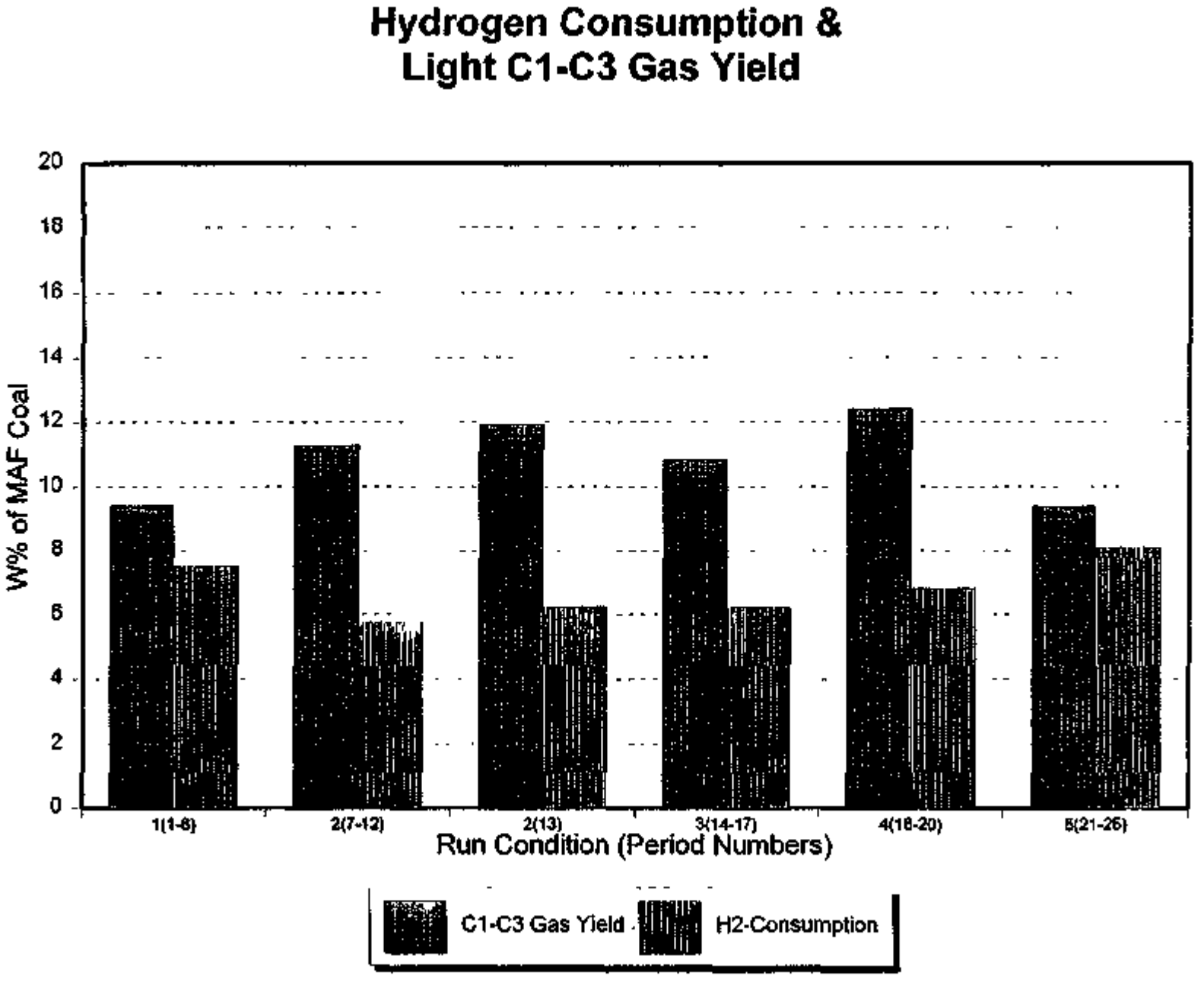

Hydrogen Consumption &
Light C1-C3 Gas Yield
W% of MAF Coal
0
2
4
6
8
10
12
14
16
18
20
1(1-6)
2(7-12)
2(13)
3(14-17)
4(18-20)
5(21-26)
Run Condition (Period Numbers)
C1-C3 Gas Yield
H2-Consumption

Figure 15

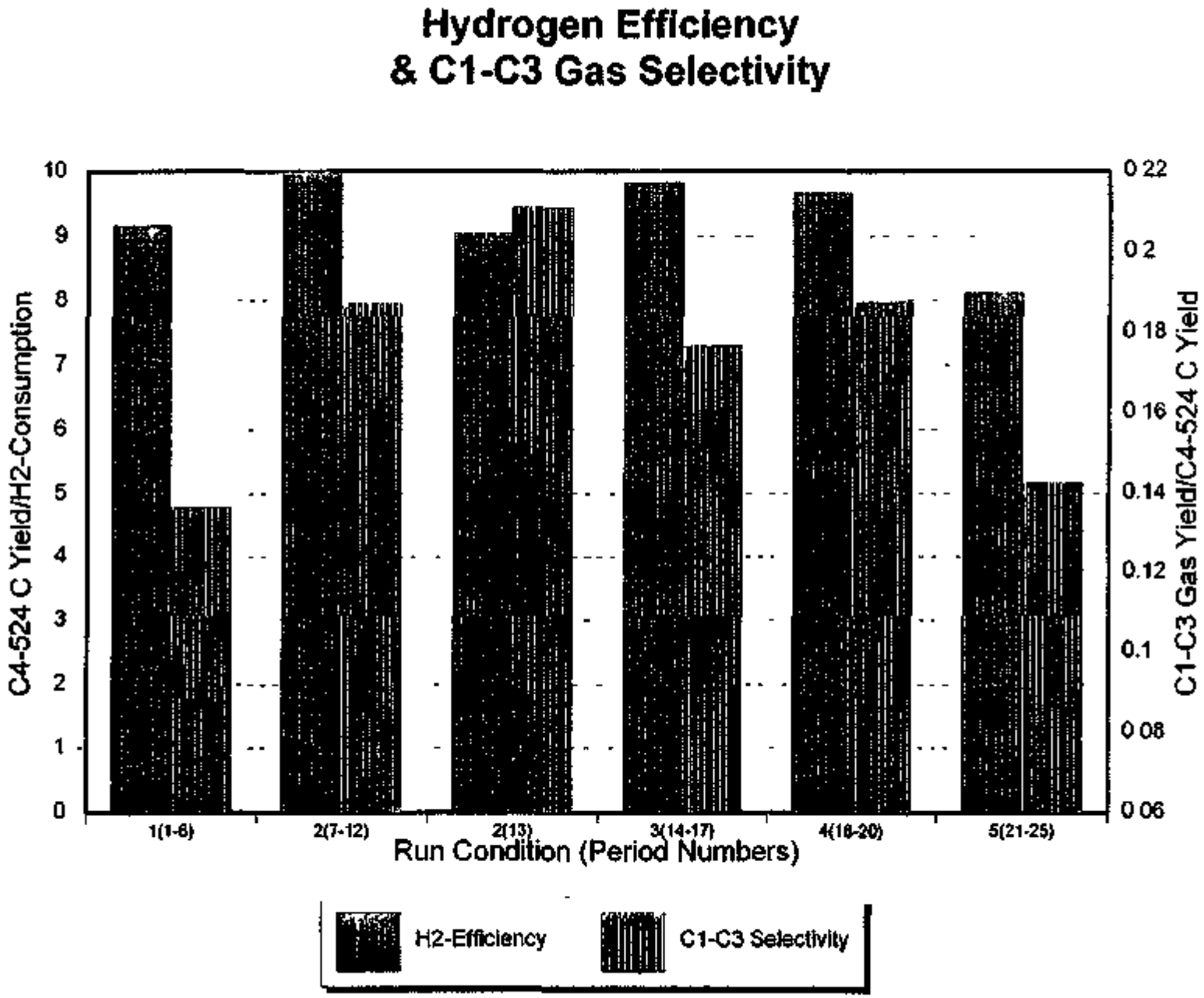
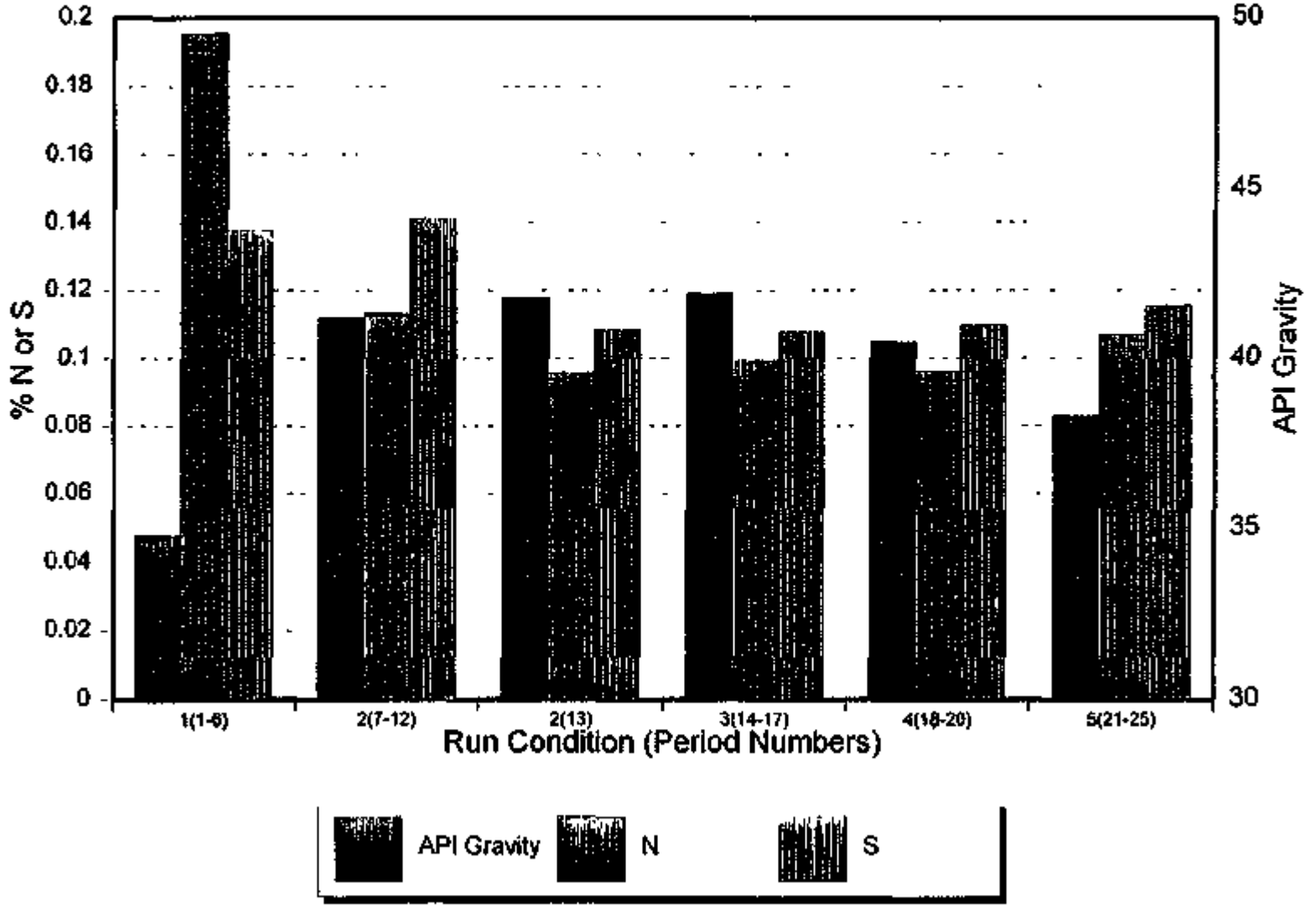

Hydrogen Efficiency
& C1-C3 Gas Selectivity
C4-524 C Yield/H2-Consumption
C1-C3 Gas Yield/C4-524 C Yield
0
1
2
3
4
5
6
7
8
9
10
0 06
0 08
0.1
0.12
0.14
0 16
0 18
0 2
0 22
1(1-6)
2(7-12)
2(13)
3(14-17)
4(18-20)
5(21-25)
Run Condition (Period Numbers)
H2-Efficiency
C1-C3 Selectivity

Figure 16

Quality of Second Stage SOH Oil



Appendix A

DATE	19-Apr-96	20-Apr-96	21-Apr-96	22-Apr-96	23-Apr-96	24-Apr-96
PERIOD	1	2	3	4	5	6
Coal Type or Bag #'s	Raw Coal	Raw Coal	Raw Coal	Raw Coal	Raw Coal	Raw Coal
FEEDS, gms						
COAL: Black Thunder	25845	33810	34770	35562	31386	35303
Was Old Agg Oil						
Wash Oil						
FF4						
FF5						
Molyvan - A	10.12	10.63	10.89	11.15	9.92	11.13
Fe - based Catalyst	2947	3097	3172	3249	2890	3242
H2S	915	909	887	903	889	1012
TNPS	2469	2667	2661	2555	2659	2723
Make-up Oil	1787	5399	6689			
VSB				2788	10253	9215
VSOH2						
VSOH1						
975F-VSOH						
ASOH						
INTERSTAGE						
SOH - OIL						
SOH - H2O						
PFL	53260	41793	42107	46626	30107	35020
PFC		2288	1904	2756	6726	8638
Water	9269	8508	8462	8567	8778	8916
Hydrogen Feed	7072	7067	7073	7068	7055	7062
TOTAL FEED	103594	105547	107736	110084	100753	111142
Products, gms						
Hydrogen in Product Gases	6076	4238	4440	4867	4596	4653
Product Gas (hydrogen & nitrogen free)	8399	8206	6936	6472	7901	6285
VSB						
VSOH2						
VSOH1						
975F-VSOH						
ASOH		4143	13929	6077	1428	
INTERSTAGE						
SOH - OIL	10444	15769	9266	8823	9035	14794
SOH - H2O	14265	19711	20138	26114	20873	18701
PFL	61620	44801	44496	45952	43402	50113
PFC	2602	4760	4357	5439	10349	13215
TOTAL PRODUCTS	103406	101629	103564	103744	97584	107761
RECOVERY, W%	99.82	96.29	96.13	94.24	96.86	96.96
FEED STREAM RATIO's (to MF Coal)						
PFL+PFC+VSB+VSOH+Makeup+Agg. Oil	2.32	1.60	1.60	1.61	1.63	1.63
IOM & ASH in PFC FEED, %		6.08	5.20	6.50	19.07	22.71
Metered H2 Cons., W% maf coal	4.46	9.77	8.85	7.22	9.07	7.92
UN-NORMALIZED GAS YIELDS, W% maf coal						
K-1 & K-2 : C1-C3 Gas Yield	15.72	15.63	11.94	10.97	14.74	9.94
K-1 & K-2 : C4-C7 Gas Yield	10.78	5.70	5.44	5.05	6.40	4.86
K-1 & K-2 : COx Gas Yield	11.15	7.02	5.94	5.21	8.00	5.84
K-1 : C1-C3 Gas Yield	7.19	5 19	4.58	4.63	5.72	4 84
K-1 : C4-C7 Gas Yield	4.65	1 92	1.70	3.19	3.39	2.84
K-1 : COx Gas Yield	10.24	5.95	5.11	4.72	7.44	5.32
Dry Coal Feed, gms	23762	30845	31697	32468	28884	32405
Calculated Coal Moisture Content, W%	8.06	8.77	8.84	8.70	7.97	8.21
Coal Conversion, SO3 free		93.38	93.99	95.14	95.44	94.96

Appendix B

DATE	25-Apr-96	26-Apr-96	27-Apr-96	28-Apr-96	29-Apr-96	30-Apr-96	01-May-96
PERIOD	7	8	9	10	11	12	13
Coal Type or Bag #'s	BAG C2	BAG C2 & C3	BAG C3 & C4	BAG C4 & C5	BAG C5	BAG C5 & C6	BAG C6 & C7
FEEDS, gms							
COAL: Black Thunder	33715	33417	29040	28109	25208	27280	27998
Wes Old Agg Oil							
Wash Oil				4892	1462		
FF4							
FF5							
Molyvan - A	10.57	10.53	9.40	9.00	8.10	8.81	9.20
Fe - based Catalyst	3079	3064	2699	2585	2324	2530	2644
H2S	828	981	960	915	847	908	872
TNPS	2639	2586	2442	2120	2107	2048	2129
Agglomerating Oil	10778	10531	9213	8825	7885	8593	9109
VSB	1614	3943	22676	21720	19524	20988	21897
VSOH2	25572		2437	2626	9815	3554	3074
VSOH1							
975F-VSOH							
ASOH							
INTERSTAGE							
SOH - OIL							
SOH - H2O							
PFL	4156	22504					
PFC	9243	12103	9157	8771	7884	8584	9084
Water	6648	6154	7241	7123	6915	7014	7651
Hydrogen Feed	7074	7076	7071	7061	7071	7074	7073
TOTAL FEED	105356	102369	92945	94757	91050	88582	91640
Products, gms							
Hydrogen in Product Gases	4610	4597	4357	4695	4803	4743	4699
Product Gas (hydrogen & nitrogen free)	6169	5677	6727	5358	5758	5167	5342
VSB			33434	27309	25421	27825	32233
VSOH2			9170	14716	19894	12628	9715
VSOH1							
975F-VSOH							
ASOH							
INTERSTAGE	304						
SOH - OIL	10054	11601	10027	8465	8485	8538	9314
SOH - H2O	19183	19067	17460	18838	15593	15720	15822
PFL	52213	45516				0	
PFC	13040	15065	11986	11693	10402	10728	12537
TOTAL PRODUCTS	105574	101523	92161	91064	91354	85349	89661
RECOVERY, W%	100.21	99.17	99.16	96.10	100.33	96.35	97.95
FEED STREAM RATIO's (to MF Coal)							
PFL+PFC+VSB+VSOH+Makeup+Agg. Oil	1.63	1.60	1.60	1.60	1.94	1.65	1.61
IOM & ASH in PFC FEED, %	17.49	24.97	18.53	18.31	21.32	20.01	18.73
Metered H2 Cons., W% maf coal	8.10	8.36	10.36	9.34	10.12	9.54	9.17
UN-NORMALIZED GAS YIELDS, W% maf coal							
K-1 & K-2 : C1-C3 Gas Yield	9.08	9.13	10.74	11.37	14.56	11.60	12.59
K-1 & K-2 : C4-C7 Gas Yield	6.17	5.41	7.03	5.23	5.25	5.38	5.22
K-1 & K-2 : COx Gas Yield	5.04	4.61	4.09	4.56	5.86	4.17	2.81
K-1 : C1-C3 Gas Yield	4.99	4.95	4.66	5.71	6.06	5.44	4.07
K-1 : C4-C7 Gas Yield	1.70	3.11	3.86	2.38	2.27	1.84	1.71
K-1 : COx Gas Yield	4.62	4 20	3.72	4.18	5.33	3.77	2.06
Dry Coal Feed, gms	31435	30653	27100	26226	23206	25280	25786
Calculated Coal Moisture Content, W%	6.76	8.27	6.66	6 70	7.94	7.33	4.33
Coal Conversion(SO3 free)	97.04	97.04	97.98	97.79	97.66	97.84	97.69

Appendix C

DATE	02-May-96	03-May-96	04-May-96	05-May-96
PERIOD	14	15	16	17
Coal Type or Bag #'s	BAG C6 & C7	BAG C7 &C8	BAG C8	BAG C8 & C9
FEEDS, gms				
COAL: Black Thunder	28383	28138	27569	27897
Wax Old Agg Oil				
Wash Oil				
FF4				
FF6				
Molyvan - A	6.56	6.51	6.40	6.44
Fe - based Catalyst	1913	1897	1867	1877
H2S	955	943	689	858
TNPS	2118	2198	2111	2195
Agglomerating Oil	9238	9145	9001	9036
VSB	22178	21994	22239	22361
VSOH2	4708	2514	2532	2597
VSOH1				
975F-VSOH				
ASOH				
INTERSTAGE				
SOH - OIL				
SOH - H2O				
PFL				
PFC	9200	9124	8981	9030
Water	8037	7995	8037	6672
Hydrogen Feed	7069	7055	7075	7075
TOTAL FEED	93805	91009	90307	89604
Products, gms				
Hydrogen in Product Gases	4696	4607	4723	4634
Product Gas (hydrogen & nitrogen free)	5951	6634	4821	5240
VSB	29516	31120	27739	28392
VSOH2	7491	7600	11242	10524
VSOH1	2438	2165	1877	1685
975F-VSOH				
ASOH				
INTERSTAGE	287			
SOH - OIL	8204	8174	6789	9449
SOH - H2O	16186	14643	16097	13867
PFL				
PFC	11426	12717	11531	11775
TOTAL PRODUCTS	86195	87660	86819	85566
RECOVERY, W%	91.89	96.32	96.14	95.49
FEED STREAM RATIO's (to MF Coal)				
PFL+PFC+VSB+VSOH+Makeup+Agg. Oil	1.67	1.60	1.62	1.63
IOM & ASH in PFC FEED, %	19.31	19.37	17.05	21.73
Metered H2 Cons., W% maf coal	9.04	9.43	9.23	9.55
UN-NORMALIZED GAS YIELDS, W% maf coal				
K-1 & K-2 : C1-C3 Gas Yield	13.16	12.02	11.73	11.22
K-1 & K-2 : C4-C7 Gas Yield	4.86	6.97	5.52	4.95
K-1 & K-2 : COx Gas Yield	4.53	4.59	1.67	4.34
K-1 : C1-C3 Gas Yield	5.55	6.01	4.18	5.96
K-1 : C4-C7 Gas Yield	1.87	5.51	1.86	2.44
K-1 : COx Gas Yield	4.08	4.52	1.26	4.08
Dry Coal Feed, gms	27123	26813	26328	26365
Calculated Coal Moisture Content, W%	4.44	4.71	4.50	5.49
Coal Conversion, SOS free	97.86	98.06	98.71	97.58

Appendix D

DATE	06-May-96	07-May-96	08-May-96
PERIOD	18	19	20
Coal Type or Bag #'s	BAG C9	BAG C9 & C1	BAG C9 & C1
FEEDS, gms			
COAL: Black Thunder	20624	18807	20122
Wax Old Agg Oil			
Wash Oil			
FF4			
FP1			
Molyvan - A	4.92	4.47	4.74
Fe - based Catalyst	1457	1291	1367
H2S	964	938	953
TNPS	1657	1508	1528
Agglomerating Oil	6930	6244	6594
VSB	17176	15497	16410
VSOH2	2345	2691	3328
VSOH1			
975F-VSOH			
ASOH			
INTERSTAGE			
SOH - OIL			
SOH - H2O			
PFL			
PFC	6930	6249	6618
Water	8742	9234	8998
Hydrogen Feed	7073	7068	7070
TOTAL FEED	73902	69532	72993
Products, gms			
Hydrogen in Product Gases	4981	5023	4874
Product Gas (hydrogen & nitrogen free)	4138	4144	4329
VSB	17809	17721	17475
VSOH2	10236	7623	6344
VSOH1	1751	3010	4572
975F-VSOH			
ASOH			
INTERSTAGE	322		
SOH - OIL	7600	7618	7926
SOH - H2O	14677	13133	12721
PFL			
PFC	8497	8852	8919
TOTAL PRODUCTS	70011	67134	67159
RECOVERY, W%	94.74	96.55	92.01
FEED STREAM RATIO's (to MF Coal)			
PFL+PFC+VSB+VSOH+Makeup+Agg. Oil	1.65	1.63	1.64
MM & ASH in PFC FEED, %	21.23	21.61	19.76
Metered H2 Cons., W% maf coal	10.69	11.26	11.32
UN-NORMALIZED GAS YIELDS, W% maf coal			
K-1 & K-2 : C1-C3 Gas Yield	10.64	13.37	12.21
K-1 & K-2 : C4-C7 Gas Yield	7.11	5.27	6.09
K-1 & K-2 : COx Gas Yield	3.36	4.21	4.01
K-1 : C1-C3 Gas Yield	5.75	6.68	6.16
K-1 : C4-C7 Gas Yield	2.57	2.64	2.51
K-1 : COx Gas Yield	3.28	4.03	3.84
Dry Coal Feed, gms	20169	18766	20078
Calculated Coal Moisture Content, W%	2.11	0.22	0.22
Coal Conversion, SO3 free	97.89	97.61	97.49

Appendix E

DATE	09-May-96	10-May-96	11-May-96	12-May-96	13-May-96
PERIOD	21	22	23	24	25
Coal Type or Bag #'s	Raw Coal	Raw Coal	Raw Coal	Raw Coal	Raw Coal
FEEDS, gms					
COAL: Black Thunder	21764	24596	25398	24964	23899
Was Old Agg Oil					
Wash Oil					
FF4					
FF6					
Molyvan - A	5.17	5.64	5.71	5.58	5.38
Fe - based Catalyst	1501	1637	1655	1618	1561
H2S	986	891	910	931	889
TNPS	1687	1857	1989	1877	1893
Agglomerating Oil					
VSB	10418	10337	7476	7493	6398
DW-HT VSOH2 (Old)	17237	19664	7094	16216	7138
DW-HT VSOH2 (Fresh)			14348	3243	12702
Wax					
DW-HT VSOH1					
DW-HT VSOH2					
SOH - OIL					
SOH - H2O					
PFL					
PFC	6875	7783	7871	7695	7420
Water	8335	8316	8107	7456	7649
Hydrogen Feed	7071	7073	7061	7067	7069
TOTAL FEED	75879	82162	81915	78566	78622
Products, gms					
Hydrogen in Product Gases	5200	5168	5193	5317	5260
Product Gas (hydrogen & nitrogen free)	4007	5203	5448	5527	4285
VSB	7456	8828	9365	8426	7259
DW-HT VSOH2 (Old)					
DW-HT VSOH2 (Fresh)					
Wax	1339	1274	1071	1518	986
DW-HT VSOH1	7176	4770	5536	6918	6519
DW-HT VSOH2	15408	16449	17300	13054	14919
SOH - OIL	8048	10262	8797	8970	9017
SOH - H2O	13837	14150	15234	14712	13051
PFL					
PFC	9260	12683	12391	11869	12307
TOTAL PRODUCTS	71731	78787	80335	76312	73602
RECOVERY, W%	94.53	95.89	96.07	97.13	96.06
FEED STREAM RATIO's (to MF Coal)					
PFL+PFC+VSB+VSOH+Makeup+Agg. Oil	1.64	1.64	1.56	1.50	1.51
IOM & ASH in PFC FEED, %	20.43	22.58	20.05	15.58	17.89
Metered H2 Cons., W% maf coal	9.46	8.82	8.42	8.06	8.62
UN-NORMALIZED GAS YIELDS, W% maf coal					
K-1 & K-2 : C1-C3 Gas Yield	10.49	12.85	12.55	13.61	9 67
K-1 & K-2 : C4-C7 Gas Yield	3.99	4.97	5.52	5.13	4 09
K-1 & K-2 : COx Gas Yield	5.79	6.27	6.47	6.83	6.46
K-1 : C1-C3 Gas Yield	7.24	5.96	6.53	5.88	6.05
K-1 : C4-C7 Gas Yield	2.36	2.20	2.23	2.07	2.45
K-1 : COx Gas Yield	5.64	5.89	6.14	6.27	6.31
Dry Coal Feed, gms	21061	23014	23643	23114	22362
Calculated Coal Moisture Content, W%	3.23	6.43	6.91	7.41	6 43
Coal Conversion, SO3 free	96.09	95.48	96.26	96.64	94.72

Appendix F: SUMMARY OF OPERATIONS

Unit Modification and Configuration

For Run ALC-1, Unit-227 was set up to process sub-bituminous coal using two 2000cc reactor stages with interstage product separation. Both stages contained no supported catalyst, using slurry catalysts mixed with the feed. Prior to the run, the system was modified to the following configuration:

- Two equal volume back-mixed reactors with internal re-circulation.
- Two liquefaction stages operated as slurry reactors at different temperatures. Dispersed Fe and Molyvan-A catalysts were used in all conditions.
- Preheater (essentially a coil preheater, 5 min residence time)
- Interstage separation and sampling system.
- Pressure filtration of CAS bottoms (O-6 bottoms).
- Vacuum still to fractionate PFL into VSOH and VSB except for Condition 1.
- Toluene extraction of PFC.
- In-line hydrotreater temperature was set at 379°C.
- The cut point of the continuous atmospheric still was set at 329°C.
- Vacuum still temperature was set at different values for different conditions (412-524°C)
- In condition 5, VSOH was dewaxed and hydrotreated prior to recycle.
- Hydrotreating of dewaxed VSOH was conducted off-line at 360°C and 15 MPa of H_2

Startup

Startup consisted of establishing the proper flows of oil and gases, presulfiding the catalyst in the hydrotreater, setting vessel temperatures, testing the first stage sample system, and increasing the reactor temperatures to 413°C. Filtered L-814, heavy gas oil, was used as a startup oil for this run. The recycle material generated during the reactor heat-up period was used to slurry the coal at the beginning of Period 1.

The startup was interrupted by a scale-like carbonaceous plug in the riser tube and bubble cap of K-1.

Upon inspection, the reactor and top head assembly were found to be clean and clear. The startup was resumed.

Shortly after gas flows were reinitiated, the first stage sample collection cylinder electrical resistance heater controller failed, allowing the heater to run continuously. This set of events caused a plug to develop in the cylinder. The cylinder was taken off the unit for cleaning while the startup resumed.

Near the end of the startup procedure, there were level control problems in the atmospheric still (CAS), which caused heavy material to get into the top of the still and overhead vessel. These fluctuations were the result of cold control-valve and pressure-sensing lines due to blocked steam tracing lines.

Condition 1 (Periods 1 through 6)

Operating Conditions, Procedures, and Changes

Period 1 started with the introduction of coal feed at 0500 hours April 19, 1996. (Each 24 hour period started and ended at 0400 hours.) The feed rate and reactor temperatures were gradually increased, reaching full coal rate of 1488 g/h at 1600 hours and Condition 1 reactor temperatures of 443°C (K-1) and 453 °C (K-2) at 2200 hours. By the middle of Period 3 the first toluene-extracted solid (ES, extracted filter cake) was available for recycle, and the full ES rate was attained in the middle of Period 5.

All of the V-100 bottoms were charged to the pressure filter. The PFL was recycled to the Unit, and the PFC was sent to the toluene-extractor. Some of the ES were recycled to the Unit.

By the end of Period 2, pressure filtrations required more than 3 hours. In Period 3, the hot separator and CAS temperatures were decreased by 14°C to 329 and 315 °C, respectively, to make the CAS bottoms less viscous. Also in Period 3, some of the CAS bottoms were added to the toluene extractor in addition to the entire PFC to fully utilize its 6000 g capacity. The IBP-399 °C cuts of the excess PFL and Extractor Oil (EO) were used to dilute the filter feed at the rate of about 15% of the CAS bottoms charge. Figure 2 presents the Downstream Product Flow Scheme at this time.

In Period 4, all the VSBs (399°C+) cut from the PFL and from the EO were recycled to the charge to meet the goal of heavy liquid extinction. Also the ES were dried for 2 hours at 182°C with flowing nitrogen to remove any residual toluene. The O-1 temperature was increased by 6°C, to 335°C.

Because of flow disruptions, causing extreme heavy oil and solids deposition throughout the column packing, the CAS was taken off line in Period 5. Additional cuts were made in the vacuum still, with the 149-316 °C fraction being pumped to the HTU (to replace the CAS overheads), the 316-399°C fraction used for pressure filter diluent, and the 399 °C+ fraction used for Unit feed. The IBP-149°C fraction of EO was removed as residual toluene.

Since the solids level in the unit had not reached a steady level by Period 5, Condition 1 was extended by 1 day to Period 6. Also, to expedite solids recycle to the Unit, the extractor cycle time was decreased from 12 hours to 6 hours.

The last filtrations of Period 6 required more than 4 hours and were discontinued to proceed with filtrations for the next condition. When the filtrations were discontinued, the unadulterated filtrate was collected before breaking the filter paper. The remaining slurry was then rerun. Most filtrations went to completion in less than 1 hour after the 2-hour charges were divided between the two filters.

Process and Equipment Operations

During Condition 1 there were occasional feed pump stoppages. Most stoppages were very brief, but the backup feed pump had to be put on line three times. Feed outages exceeded 60 minutes twice during Period 5.

There were O-1 level control problems through Period 5. The level was erratic, sometimes causing decreased unit back pressure and excessive flow downstream to the O-3 and CAS. The control valves were not dirty, but they did not pass a steady flow until a larger, "G" trim was installed. Initial problems with the O-1A level were alleviated when plugged steam tracing lines were cleared.

The CAS overflowed due to the flow surges from O-1 and O-3 and also when the discharge line plugged. Additionally, the CAS was off-line for 35 minutes after the relief valve on the circulating pump stuck open, losing 1 gallon of slurry. With the reliability of the CAS hindered by these problems, it was taken off line in Period 5 for the remainder of the run.

Operations in the workup Period 6 were smooth except for a failed electric resistance heater in the hydrotreater. Its temperature decreased by 110°C for 4 hours until it was repaired.

Condition 2 (Periods 7-13)

Operating Conditions, Procedures, and Changes

In Condition 2, the coal feed was changed from raw coal to coal previously cleaned by oil agglomeration at CONSOL, Inc. (HTI-6455). The 23% oil content of this coal necessitated a reduced oil recycle rate and an additional VSOH product. Accordingly, all PFL and EO was vacuum-distilled. For EO, fractions of IBP-149 °C (toluene to storage), 149-316 °C (filter diluent), and 316°C+ (Unit feed) were collected. PFL was fractionated into VSOH (413° C-, storage and buffer oil) and VSB(413 °C+, recycled). The PFC was recycled without toluene extraction. The original hourly feed blend was:

Coal (HTI-6455)	1953 g
PFC	535 g
VSB (524°C+ from PFL)	671 g

VSOH (524°C- from PFL)	272 g
VSB (316°C+ from EO)	100 g

These feed rates were attained at 1400 hours on Period 7 when the vacuum still products became available. However, the viscosity of the above feed blend was too high and it required more than 10 hours to obtain a uniform slurry feed blend. As a result, the cut point of vacuum still was lowered to ease the feed blending Starting from Period 8 only two distillation fractions were collected from the PFL in vacuum still, with the cut point initially set at 371°C. This Downstream Product Flow Scheme is shown in Figure 3. The hourly feed blend was changed to:

Coal (HTI-6455)	1627 g
PFC	380 g
VSB(413°C* from PFL)	916 g
VSB (316°C+ from EO)	25 g

The vacuum still cut point was increased to 385 °C in Period 9, to 413 °C in Period 10, and to 427°C in Period 11. Also in Period 11, the O-1 temperature was increased from 335 to 343 °C. In Period 12, in response to a very viscous feed (up to 3000 cp at 116°C) and feed pumping problems, 100 g/h VSOH was added to the charge for 5 hours until VSB at a lower, 413°C, cut point became available at 1300 hours. Therefore, it was never possible during Condition 2, even Conditions 3 and 4, to remove distillate stream with a boiling range (343-524°C) equivalent to the foreign agglomerating oil used to prepare the agglomerated coal feed.

Condition 2 was extended for one day in order to obtain a smooth work-up period.

Process and Equipment Operations

There were numerous feed stoppages during Condition 2, with extended feed outages (greater than 10 minutes) in Periods 7, 10, and 11. The stoppages were usually the result of problems at the feed pump checks, but others were due to loss of slurry circulation. Wash oil was added when necessary to regain flow, and buffer oil (VSOH) was used to maintain reactor feed during the extended feed outages. (Wash oil was L-814 until 1100 hours of Period 11 when the wash pot was drained and filled with run-generated VSOH) Feed stoppages became less frequent at the end of Period 12 after the P-2 temperature was decreased from 127 to 116°C to more closely match the slurry preparation temperature.

Condition 3 (Periods 14 through 17)

In Condition 3 the catalyst addition rate was decreased from 112 to 79 g/h for the iron catalyst and from 0.39 to 0.27 g/h for the Molyvan A catalyst. An additional PFL cut at 343°C was made with the vacuum still As shown in the Downstream Product Flow Scheme, Figure 4, the IBP-343°C fraction of the PFL ($VSOH_1$) was removed as product and the 343-413 °C fraction ($VSOH_2$) was

used as buffer oils and diluent when needed. Starting from Period 15, the vacuum still cut point was increased by 5 °C to make the VSB IBP closer to 413 °C and to decrease the net VSB yield.

There were occasional feed stoppages in every period of condition 3. The stoppages were usually very brief, and none were longer than 10 minutes.

In Period 15, the slurry feed circulator and HTU feed pump were taken off-line for repacking. In Period 17 the water pump for the O-1A separator was cleaned and rebuilt. Also, the level control valve for the O-1 separator stuck open, causing the unit back-pressure to decrease by 350 psig with no impact on unit operations.

Condition 4 (Periods 18 through 20)

In condition 4 the space velocity was decreased from 561 to 401 kg dry coal/h/m^3(ea. reactor). Accordingly, the hourly feed rates were:

Oil Agglomerated Coal (HTI-6455)	1162 g
PFC	271 g
VSB from PFL (412 °C)	654 g
EO VSB (343°C+)	18 g
Fe Catalyst	56 g
Molyvan-A	0.19 g
TNPS	64 g

These feed rate changes were completed by 1100 hours of Period 18.

The O-3 safety relief valve was replaced in Period 18 when it was leaking vapors, the feed slurry circulation pump packing was tightened in Period 19 after a significant leak, and the coupling for the HTU pump was replaced in Period 19.

Condition 5 (Periods 21 through 25)

Operating Conditions, Procedures and Changes

In Condition 5, the coal feed was changed back to the raw coal at 1068 gm/hr. The recycle material included dewaxed, hydrotreated VSOH (343 °C+). Figure 5 presents the Downstream Product Flow Scheme for Condition 5. The dewaxing and hydrotreating steps were performed as previously described for preparing the initial DW-HT VSOH, except that the hydrotreater was operated at 360°C with a feed rate of 650 g/h. The other recycle components were PFC (320 g/h), 524 °C+ solids-free VSB (437 g/h), and 371 °C+ EO VSB (20 g/h). These feed changes were completed by 1100 hours Period 21.

The downstream processing required about two days to complete. As a result, preprepared DW-HT VSOH from previous runs with Black Thunder coal was used until the material from Condition 5 became available during Period 23. This material was also used in most of Period 24 to save the DW-HT VSOH for the work up Period 25. However, because of prolonged pressure filtration times, there was still not enough DW-HT VSOH produced during the run for the entire Period 25.

Starting from Period 22, there were insufficient VSB available for the specified recycle. All VSBs produced were recycled, and DW-HT VSOH was recycled at a rate to maintain the total VSB-plus-DW-HT VSO at 1102 gm/hr. Also starting from Period 23, the DW-HT VSOH was substituted for the EOVSB since it was not available. In Period 24, the sum of VSB and DW-HT VSOH was decreased to 1050 gm/hr to account for wax yield and 343°C- rejected.

Process and Equipment Operations

At the beginning of Period 21 there were difficulties in maintaining unit feed. Vaporization of light material in the circulating lines were partly responsible. During the rest of Condition 5 there were occasional, very brief feed stoppages.

In Period 22 the upper heater of the in-line hydrotreater failed. The middle temperature decreased by about 93 °C for 3 hours until it was replaced.

On-Line Time Summary

The chronology of the operations for Run ALC-1 is listed in Table 32 along with a summary of the total time in startup, run, shutdown, and down modes. Unit 227 operated for 694 hours with one interruption during startup for an On-Line Efficiency of 97.2 %.

Shutdown and Inspection

Shutdown commenced at the end of Period 25 (0400 hours May 14, 1996). It went through very smoothly. All of the process equipment was in order; only routine maintenance was required

Appendix G. Boiling Point Distribution of First and Second Stage Overheads

	Temperature [degree F] First Stage Overheads					Temperature [degree F] Second Stage Overheads						
Vol% / Period	6	13	17	20	25	5	6	12	13	17	20	25
0.5	130	123	99	137	98	44	37	41	84	67	97	47
5.0	209	207	198	217	188	175	183	165	174	172	178	171
10.0	264	259	250	266	240	201	243	190	201	196	209	200
15.0	286	275	269	290	268	228	274	215	225	220	239	228
20.0	305	291	287	315	288	254	304	241	249	244	264	257
25.0	326	307	305	339	309	281	335	264	267	265	273	270
30.0	347	322	322	364	329	307	365	272	275	272	286	280
35.0	367	338	340	388	349	334	393	282	284	281	311	292
40.0	394	372	358	415	385	360	420	297	303	295	342	323
45.0	423	396	386	443	418	394	443	324	329	322	372	357
50.0	451	423	415	469	453	428	465	350	360	349	402	393
55.0	479	451	445	493	487	458	487	386	391	385	433	430
60.0	505	481	474	518	519	488	508	418	428	417	461	467
65.0	532	510	503	543	548	515	528	452	459	450	491	501
70.0	559	543	536	569	571	540	547	487	494	486	521	532
75.0	584	580	570	599	596	566	567	525	530	523	552	561
80.0	616	629	615	637	624	592	589	563	567	563	582	588
85.0	652	675	664	671	652	625	617	606	613	607	624	620
90.0	687	718	707	706	680	665	653	664	670	666	671	656
95.0	734	769	759	755	720	712	698	726	733	729	729	698
99.5	832	860	848	846	804	813	799	827	836	829	828	787

Appendix H: Composition of SOH Water Products from Run ALC-I

SOH Water Sample Source			Elemental Composition, wt % of Sample		
Period	Stage	pH	S (a)	N (b)	C (b)
6B	1st	9.22	0.79	1.52	1.72
13B	1st	9.07	1.24	1.77	1.84
13B	2nd	9.74	2.69	2.14	0.54
17B	1st	9.19	1.07	1.43	1.69
17B	2nd	9.21	2.50	2.00	0.27
20B	1st	9.02	1.05	1.46	1.70
20B	2nd	9.29	2.52	1.85	0.12
25B	1st	8.94	0.66	1.25	1.71
25B	2nd	9.09	2.22	1.63	0.15

(a) Total S analysis by LECO SC-32 Sulfur Analyzer
(b) C and N analysis by LECO CHN-1000

Note: Analysis Performed by CONSOL, Inc

Appendix I: Impact of Carbon Loss in SOH Water on Distillate Yield

	Original	New (C in Sour Water)
DATE	05/08/96	05/08/96
PERIOD	20	20
Coal Type or Bag #'s	C9 & C1	C9 & C1
CONDITION	4	4

NET NORMALIZED YIELDS, W% maf coal

C1 in Gases	3.32	3.31
C2 in Gases	3.49	3.48
C3 in Gases	5.59	5.57
C1-C3 in Gases	12.40	12.35
C4-C7 in Gases	6.48	6.46
IBP-177 C (IBP-350 F)	16.53	17.00
177-260 C (350-500 F)	9.70	9.81
260-343 C (500-650 F)	23.88	23.79
343-454 C (650-850 F)	13.24	12.94
454-524 C (850-975 F)	-3.75	-3.62
524+ C (975+ F)	4.56	4.40
Unconverted Coal	2.51	2.51
Water YLD by Material Balance	16.20	15.68
COx	4.27	4.90
NH3	0.90	0.90
H2S	-0.09	-0.08

PROCESS PERFORMANCE, W% maf coal

Hydrogen Consumption	6.8	6.8
Coal Conversion (SO3 Free)	97.5	97.5
524+ C Conversion	92.9	93.1
C4-524 C Distillates	66.1	66.2
IBP-343 C Distillates	50.1	50.6
Desulfurization	78.6	78.7
Denitrogenation	70.9	71.3
524+C Resid Yield	4.6	4.4
WAX Product		

PROCESS CONDITIONS

Space Velocity, maf coal / reactor volume		
K-1 (lb/hr / ft3)	26.1	26.1
K-2 (lb/hr / ft3)	26.1	26.1
Reactor Temperatures, F		
K-1	828.9	828.9
K-2	846.3	846.3
Fresh Catalyst Concentration, metal wppm maf coal		
Mo in Molyvan A	68	68
Fe in Fe-based	6811	6811

APPENDIX J: Properties of Recycle Solvents and PFC (daily analysis)

Period Date	1 4/19	2 4/20	3 4/21	4 4/22	5 4/23	6 4/24	7 4/25	8 4/26	9 4/27	10 4/28	11 4/29	12 4/30	13 5/1
PFL:													
IBP, °C		245	239	253	218	218		211					
IBP-343		8.44	10.15	9.91	13.34	17.43		15.27					
343-454		51.38	47.69	47.32	47.09	40.47		45.16					
454-524		14.50	14.23	14.11	12.26	12.65		14.63					
524C*		24.95	27.29	28.21	26.68	28.91		24.31					
Loss		0.73	0.64	0.45	0.63	0.54		0.63					
VSOH:													
IBP,°C								221	218	219	227	220	226
IBP-343								63.7	68.31	51.5	36.55	37.33	53.8
343-454								35.4	30.98	47.7	62.35	61.98	45.1
454-524								0.7	0.7	0.8	0.7	0.4	0.9
524C+								0.0	0.0	0.0	0.0	0.0	0.0
Loss								0.2	0.0	0.0	0.4	0.29	0.2
VSB													
IBP,°C								352	390	368	403	393	373
IBP-343								0.0	0.0	0.0	0.0	0.0	0.0
343-454								51.37	44.54	41.44	30.91	34.20	41.63
454-524								16.80	21.14	17.90	21.44	22.48	19.11
524 C*								31.39	34.06	40.23	47.14	42.81	38.91
Loss								0.44	0.26	0.0	0.51	0.51	0.35
QI on PFCs	71.09*	81.94*	86.62*	76.53*	81.89*	85.21*	59.50	63.23	54.85	61.43a	62.76	58.94	55.24
ASTMash	37.94	57.88	59.92	56.03	61.52	63.44	43.97	45.94	42.59	44.99	47.04	46.04	40.97
S in ash	—	9.50	9.81	9.82	9.66	10.0	7.62	5.33	3.01	5.01	4.48	3.22	3.0

Period Date	14 5/2	15 5/3	16 5/4	17 5/5	18 5/6	19 5/7	20 5/8	21 5/9	22 5/10	23 5/11	24 5/12	25 5/13
DW-HT												
VSOH1												
IBP,°C				203			227	185	142	191	197	181
IBP-343				82.85			54.25	75.44	83.1	85.30	70.52	64.27
343-454				16.94			45.65	23.5	16.9	11.7	29.27	33.13
454-524				0.0			0.0	1.06	0.0	2.95	0.21	2.60
524C+				0.0			0.0	0.0	0.0	0.0	0.0	0.0
Loss				0.21			0.1	0.0	0.0	0.0	0.0	0.0
DW-HT												
VSOH2:												
IBP,°C		263	245	245		243	262	305	274	330	333	318
IBP-343		28.73	35.21	35.90		23.66	9.71	5.1	14.9	5.12	5.17	10.2
343-454		70.80	63.41	63.12		75.66	90.0	86.7	75.3	76.7	77.6	81.6
454-524		0.0	0.89	0.69		0.68	0.0	6.53	7.94	16.4	14.52	5.55
524C+		0.0	0.0	0.0		0.0	0.0	1.57	1.86	1.47	2.03	2.55
Loss		0.39	0.49	0.29		0.0	0.39	0.0	0.0	0.29	0.68	0.1
VSB												
IBP,°C		388	380	373		397	389		509	497	507	
IBP-343		0.0	0.0	0.0		0.0	0.0		0.0	0.0	0.0	
343-454		38.99	39.86	39.20		33.68	36.58		0.0	0.0	0.0	
454-524		22.70	21.68	18.29		22.22	18.33		7.54	10.25	7.71	
524C+		38.04	38.20	42.07		43.58	44.74		91.97	89.34	91.72	
Loss		0.27	0.26	0.44		0.52	0.35		0.49	0.41	0.57	
QI on PFC	56.92	60.34	61.66	63.45	61.85	64.88	59.96	62.58	66.78	60.21	46.79	53.92
ASTM ash	41.71	43.05	42.06	43.06	43.72	45.02	41.78	43.76	4.26	42.70	34.38	40.15
S in ash	2.47	3.57	3.28	3.41	3.92	3.35	3.29	2.85	7.46	9.26	9.30	10.80

*Toluene extracted PFC

Appendix K: SPECIAL SAMPLES FOR CONSOL INC.

COLLECTION PERIODS - 6, 13, 17, 20, & 25

SAMPLE STREAM	AMOUNT, GM
Hydrotreated SOH Oil (2nd stage)	2 gal
SOH water (2nd stage)	250
1st stage SOH (Collected immediately after W-up period)	50
ASOH	50
PFL (period 5 only)	150
PFC (toluene extracted)	150
PFC	50
VSOH(from PFL)	100
VSB(from PFL)	100
Dewaxed VSOH	100
Wax	350
Hydrotreated Dewaxed VSOH	100
CAS BOTTOMS (all W-up periods)	150
FEED SLURRY (all W-ups)	200
INTERSTAGE SLURRY SAMPLE(after W-up)	150
L-814 (once per run)	200

SECTION THREE

LDP Associates

LDP ASSOCIATES

Michael Peluso, Proprietor **32 Albert E. Bonacci Dr.**
609-586-2301 **Hamilton Square, N.J. 08690**

November 12, 1996

Dr. Ed Givens
Center for Applied Energy Research
3572 Iron Works Pike
Lexington, Kentucky 40511-8433

Dear Ed:

Subject: QUARTERLY PROGRESS REPORT: JULY THRU SEPTEMBER 1996

For the quarter ending September 30, 1996 the following subcontract services (UKRF-4-25582-92-75) were performed in support of the DOE Advanced Concepts Program (DE-AC22-91PC91040):

ALC-1 ANALYSIS

The elementally balanced material balance procedure was used to calculate estimated yields, hydrogen consumption and distillate boiling range yields for Period #12 of Condition #2. As expected C_{4+} distillate yield increased significantly versus the original Condition #2 material balance Period (#13). This preliminary result was later confirmed by HTI's similar finding.

The analysis of the Separator Overhead Water (SOH) samples for the five conditions was received from CONSOL and reviewed. A method was developed for incorporating these analysis into HTI's normalized material balance, but an assumption regarding the inorganic carbon content of the SOH was also required. The method was discussed with both CONSOL and HTI and was used to provide a fully normalized HTI material balance for one of the conditions. The effect of including the SOH water in the normalized balance was not significant.

Based on metals analysis performed by CONSOL a molybdenum balance was calculated for Conditions #2 thru #5 of the run. The results indicate the moly leaving the unit was much greater than the reported moly input. The reason for this apparent inconsistency is being investigated.

Comments on HTI's draft run report were prepared and transmitted. A request was made for the preparation, in the future, of a simplified but comprehensive printout of the input and output of HTI's normalized material balances.

ALC-2 RUN PLAN

The first draft of the run plan was reviewed and comments were discussed with Ed Givens. A suggestion was made to lower the recycle solvent to

coal ratio to approximately 1.8/1 for the high solids recycle periods. The revised backend distillation system was also reviewed.

A subsequent second draft of the run plan was reviewed and comments were discussed with CONSOL and CAER.

REACTOR MODELING

In response to questions raised by Prof. Grulke (U of K), an information package was prepared to assist in the development of the above ASPEN based model. Questions on resid properties, material balance methods and reactor operating parameters & yields were addressed.

ECONOMIC ASSESSMENT

No activity.

Very truly yours,

Michael Pelusо
LDP Associates

cc: F. Derbyshire @ CAER
R. Anderson @ CAER
R. Winschel @ CONSOL
T. Lee @ HTI

SECTION FOUR

CONSOL, Inc

February 6, 1997
Report Period: July 1 - September 30, 1996

SUBCONTRACT TITLE AND NUMBER:
Subcontract UKRF-4-25582-92-76 to CONSOL Inc.
Under DOE Contract No. DE-AC22-91PC91040, "Advanced Coal Liquefaction Concepts for the PETC Generic Bench-Scale Unit"

SUBCONTRACTOR NAME:
CONSOL Inc.
Research & Development
4000 Brownsville Road
Library, PA 15129

SUBCONTRACT PERIOD: November 26, 1991 - February 28, 1997 (future extension to September 30, 1998, expected)

PRINCIPAL INVESTIGATORS: R. A. Winschel, G. A. Robbins

SUBCONTRACT OBJECTIVES: No change.

SUMMARY OF TECHNICAL PROGRESS - OVERALL

Most of the activities this quarter were directed toward evaluation of Run ALC-1 results and planning for Run ALC-2. The process oil samples collected during Run ALC-1 were characterized by CONSOL under DOE Contract DE-AC22-94PC93054. Analyses showed that about 1% of the feed coal carbon reports to the separator overhead water product. It appears that any error resulting from omitting this stream from elemental balance calculations should be small. There had been concern that significant amounts of light oils were recycled in Run ALC-1. However, the analysis of feed slurry samples showed that they contained less than 5% 343 °C⁻ (650 °F⁻) material. The concentrations of Mo and Fe in feed slurry and pressure filter cake ash samples were used to address catalyst issues within Run ALC-1. The Mo and Fe concentration results were combined with HTI material balance data for Run ALC-1 to calculate Mo and Fe balances and determine the apparent addition rates of Mo and Fe catalysts. The resulting Mo and Fe material balance closures were between 80-120%. The results indicate that the Mo addition rate was inadvertently 10-169% higher than the target rate. The highest Mo addition rate was during agglomerated coal feed periods. The results show a low Fe addition rate in Period 13 (39% of the target rate), and 4% to 36% higher than design rate in the other periods. The analysis of vacuum distillates from Run ALC-1 indicate that the bench-unit dewaxing operations

effectively removed paraffins from the vacuum distillate. However, the low concentration of wax in the feed minimized the impact of dewaxing on solvent quality improvement. The solvent hydrotreating operation effectively increased the hydroaromatic hydrogen content of the distillate and improved its solvent quality. The wax product from Run ALC-1 was characterized; it has a purity of about 75%. CONSOL participated in planning for Run ALC-2. A paper on the results of Run ALC-1 was presented at the DOE Contractors Review Conference and at the Thirteenth Annual International Pittsburgh Coal Conference.

Activities planned for next quarter include completion of the elemental and material balances for the agglomerate production runs, planning and on-site support for Run ALC-2, and initial planning for Run ALC-3.

SUMMARY OF TECHNICAL PROGRESS - BY TASK

Task 2 - Laboratory Support

The process oil and water samples collected during Run ALC-1 are being characterized by CONSOL under DOE Contract DE-AC22-94PC93054.

Analysis of Separator Overhead (SOH) Water Samples From Run ALC-1

The analyses of Run ALC-1 SOH water samples were completed. Samples from both Stage 1 and Stage 2 were analyzed for pH and weight percent C, N, and S (Table 1) for HTI's elemental balances. Relative to the Stage 1 samples, the Stage 2 samples had generally higher pH and S and N concentrations, and lower C concentration. At the SOH water product flow rates in Run ALC-1 (about 14 kg/day), the carbon content in the water is about 210 g/day (depending on the production split between the two stages). At the coal feed rate of Run ALC-1 (about 26 kg/day), the coal carbon feed rate is about 16.6 kg/day. Based on this analysis, about 1 wt % of the coal carbon and somewhat greater proportions of the coal sulfur and nitrogen report to the SOH water. It appears that any error resulting from omitting this stream from elemental balance calculations should be small.

The phenolic compounds in one SOH water sample (Period 13B, first stage) were non-quantitatively characterized by GC/MS analyses. The procedure used was acidification of the sample with HCl from a pH of about 9 to a pH of about 1 and extracton of the tar acids with methylene chloride. The appearance of the liquid changed from clear, bright yellow to colorless

and slightly turbid upon acidification. Odors detected during acidification (in chronological order) included H_2S, SO_2, and phenols. Only phenol and the cresols were identified in the tar acids.

Determination of Light Oil Contents of Feed Slurry Samples from Run ALC-1

There had been concern that significant amounts of light oils were recycled during Run ALC-1, due to inefficient distillation and other operating conditions. To address this concern, feed slurry samples from each condition of Run ALC-1 were distilled to 343 °C (650 °F) and 524 °C (975 °F). The resid was extracted with tetrahydrofuran, and ashed to determine other components as shown in Table 2. An important result from this characterization is that only a small quantity of 343 °C⁻ (650 °F⁻) material was recycled in Run ALC-1 (≤5% of feed slurry). This presumes that no light material was lost from the samples which were distilled by CONSOL.

Determination of Dispersed Catalyst Concentrations in Run ALC-1

Mo and Fe concentrations in the second-stage pressure-filter cake (PFC) samples from Run ALC-1 were determined. They are presented in Table 3 on three bases: (1) metal oxide as weight percent of the SO_3-containing ash; (2) element as a weight percent of the SO_3-free ash; and (3) element as a weight percent of the SO_3-containing ash. The CONSOL Mo values are in good agreement with those reported by HTI for the second-stage pressure-filter cake. However, many of CONSOL's Fe values are 20% or more higher than those reported by HTI.

Mo and Fe concentrations were also determined in the 524 °C⁺ (975 °F⁺) resids from all of the Run ALC-1 feed slurry samples (Table 4). The data provide a direct measurement of the total Mo and Fe concentrations in the reactor feed. The concentrations of the metals in the ash agree well with the pressure filter cake (PFC) results reported in Table 3. Elemental Mo concentrations for the entire set (PFC and feed slurry) ranged from 0.19 to 0.47 wt % of the SO_3-free ash, and approximately doubled when agglomerated coal was fed (partly in response to the reduced ash content of the agglomerated coal). Fe concentrations for the entire set ranged from 17.9 to 28.1 wt % of the SO_3-free ash, and decreased from Condition 2 through the end of the run.

The Mo and Fe concentration results were combined with HTI material balance data to calculate Mo and Fe balances and determine the apparent addition rates of Mo and Fe catalysts. Intermediate and final calculated results are shown in Table 5. The following assumptions were used in the calculations: raw coal SO_3-free ash content of 5.50% MF; agglomerated coal SO_3-free ash content of 3.30% MF; added Fe_2O_3 was 1.4% or 0.98% of the MF coal rate (for calculating

total ash fed to system); Mo in the raw or agglomerated coal was ignored (the MF raw or agglomerated coal contains ca. 2 ppm Mo); the Fe_2O_3 content of the raw coal SO_3-free ash was 6.66%; and the Fe_2O_3 content of the agglomerated coal SO_3-free ash was 9.57%. These data were obtained from this report, the April-June quarterly report for this contract, and HTI's run report.

The Mo and Fe balances were calculated for each of the periods shown as the ratio of total grams of Mo or Fe out to the total grams of Mo or Fe in. Total Mo or Fe in was obtained by multiplying the weight percent Mo or Fe in the feed slurry SO_3-free ash by the grams of ash fed in that period in the fresh coal and fresh iron catalyst, and in the pressure-filter cake (PFC) recycle. The amount of ash fed in the fresh coal was obtained from the amount of MF coal fed (HTI material balance data) and the SO_3-free ash content of the uncleaned or agglomerated coal. The ash fed in the added Fe catalyst was obtained as 1.4 wt % of the amount of MF coal fed (for the 10,000 ppm Fe target addition rate), or as 0.98 wt % of the amount of MF coal fed (for the 7,000 ppm Fe target addition rate). The ash fed in the recycle PFC was obtained from HTI's recycle ratio of insoluble organic matter (IOM) plus SO_3-free ash to MF coal, and HTI's measurements of IOM and SO_3-free ash contents of the PFC. The total amount of Mo or Fe out was obtained by multiplying the wt % Mo or Fe in the PFC by the grams of total PFC produced for the period (HTI material balance data).

Apparent catalyst addition rates were obtained by subtracting the amount of Mo or Fe in the recycled PFC from the total amount of Mo or Fe fed in the feed slurry. The rates then were put on an MF coal basis. In the Fe case, the coal Fe contribution was backed out, so that the results represent only added Fe. The ca. 2 ppm Mo in the raw coal was not backed out.

The resulting Mo balances were 80-119%, and the Fe balances were 98-120%. These indicate that the balances were generally good; that is, there was not a consistent or gross imbalance between feed and product rates. These balances generally verify the validity and consistency of the analytical measurements. The calculated apparent Mo catalyst addition rates (MF coal basis) were 110-269 ppm Mo when the target Mo concentration was 100 ppm, and 94-161 ppm when the target rate was 70 ppm Mo. The calculated apparent Fe catalyst addition rates (MF coal basis) were 3,928-13,642 ppm Fe when the target Fe concentration was 10,000 ppm, and 7,309-8,571 ppm when the target rate was 7,000 ppm Mo.

These results show that the Mo added was 10-169% in excess of the target rate. The amount in excess seemed to be largest during periods when agglomerated coal was fed. The generally good Mo balance suggests that the Mo addition rate was inadvertently high, rather than being caused by introduction of Mo from a spurious source. Any spurious source of Mo would have to introduce a high Mo concentration in the feed slurry to be consistent with these results. The results show that the Fe added in Period 13 (Condition 2) was only 39% of the target rate, but in the other periods was 4-36% in excess of the target rate.

Effectiveness of Dewaxing and Solvent Hydrotreating in Run ALC-1

Vacuum distillates from Run ALC-1 were dewaxed in the laboratory and the dewaxing feeds and fractions were analyzed (microautoclave solvent quality and NMR) to determine the effectiveness of the Run ALC-1 dewaxing and hydrotreating operations. The vacuum distillates tested were either vacuum still overhead (VSOH) samples or the laboratory-generated 454 °C⁻ (850 °F⁻) distillate of O-6 bottoms samples.

Table 6 shows that the O-6 bottoms distillate contained about 5-6% wax before dewaxing operations were started (Period 20). During operations with dewaxing (Period 25), the wax content of the second-stage heavy distillate (VSOH) product was reduced to 1-2%, presumably mostly freshly produced wax. The heavy distillate (VSOH, dewaxed) also taken from period 25 but downstream of dewaxing was virtually devoid of wax.

The ^{1}H-NMR proton distributions and microautoclave solvent quality test results of heavy distillates relevant to the dewaxing operations are shown in Table 7. The data are summarized in Table 8; averages and standard deviations are shown where possible. The data in Table 8 show that the paraffinic nature of the heavy distillate was reduced by dewaxing and there was a minor improvement in solvent quality, as measured by two types of microautoclave liquefaction tests. Hydrotreating was effective in producing hydroaromatics and in improving solvent quality.

In summary, the results indicate that the bench-unit operations were quite effective in dewaxing the vacuum distillate. However, the low concentration of wax in the feed limited the impact of the dewaxing on improving solvent quality. The solvent hydrotreating operation increased the hydroaromatic hydrogen content of the distillate and improved its solvent quality.

Analysis of Wax Produced in Run ALC-1

The wax produced in period 25 of Run ALC-1 was characterized by elemental, ^{1}H-NMR, and GC/MS analyses (Table 9). The wax was black and had a strong odor. The elemental analysis indicates that it contained over 13% hydrogen; however, four replicate elemental analyses gave poor repeatability (about 5% relative). We assume that the reported carbon content (74 wt %) is incorrectly low, because the ^{1}H-NMR and GC/MS analyses did not indicate major concentrations of any components except alkanes. The wax was de-oiled via our standard dewaxing procedure at -5 °C in acetone to provide an indication of the wax purity. This yielded 75% de-oiled wax, but also 2% THF insolubles (Table 9). The de-oiled wax has less than 1% aromatic protons (Table 9).

Preparation For Run ALC-3

A sample of each catalyst precursor which will be tested in Run ALC-2 was requested from UK/CAER. The sample will be used to test methods to integrate catalyst addition during oil agglomeration in Run ALC-3. Summaries of the catalyst addition techniques used by CAER, including those used in Run ALC-2, were requested.

Task 3 - Continuous Operations

CONSOL reviewed and commented on the drafts of the ALC-2 run plan. CONSOL reviewed and approved the HTI draft flowsheet for the continuous vacuum and atmospheric distillation and batch solids extraction systems for Run ALC-2. G. Robbins met with E. Givens (UK/CAER) and T. Lee (HTI) to discuss issues related to the Run ALC-2 run plan, including: recycle solids concentration; catalyst type and concentration; the distillation system; addition of agglomeration or solvent hydrogenation to the run plan; and the potential advantage of adjusting the hydrogen feed rate if the space velocity is adjusted (i.e., fixing the ratio of hydrogen feed rate to MF coal feed rate). The run plan was discussed with M. Peluso (LDP Associates).

Task 4 - Technical Assessment

The CONSOL portion of the draft quarterly Technical Progress Report for January through March was revised as requested by DOE and resubmitted to UK. A report summarizing the Run ALC-1 post-run meeting at Lawrenceville, NJ, on June 16 was issued to the project participants. CONSOL's comments on HTI's draft Run ALC-1 report were discussed with T. Lee of HTI.

The paper "Testing of Advanced Liquefaction Concepts in HTI Run ALC-1: Coal Cleaning and Recycle Solvent Treatment" was presented by G. A. Robbins at the DOE First Joint Power and Fuel Systems Contractors Conference and at the Thirteenth Annual International Pittsburgh Coal Conference. An electronic copy of the paper was sent to the Center for Conference Management, at DOE's request. Presentations were prepared on Runs ALC-1 and ALC-3 for the planning meeting in early October.

R. A. Winschel
Project Manager

/s

TABLE 1

COMPOSITION OF SOH WATER PRODUCTS FROM HTI RUN ALC-1

SOH Water Sample Source			Elemental Composition, wt % of Sample		
Period	Stage	pH	S (a)	N (b)	C (b)
6B	First	9.22	0.79	1.52	1.72
13B	First	9.07	1.24	1.77	1.84
13B	Second	9.74	2.69	2.14	0.54
17B	First	9.19	1.07	1.43	1.69
17B	Second	9.21	2.50	2.00	0.27
20B	First	9.02	1.05	1.46	1.70
20B	Second	9.29	2.52	1.85	0.12
25B	First	8.94	0.66	1.25	1.71
25B	Second	9.09	2.22	1.63	0.15

(a) Total S analysis by LECO SC-32 Sulfur Analyzer
(b) C and N analysis by LECO CHN-1000 Analyzer

TABLE 2

CONCENTRATION OF COMPONENTS OF THE FEED SLURRY SAMPLES FROM HTI RUN ALC-1

Date, 1996	4/23	4/24	5/1	5/5	5/8	5/13	5/5(a)
Period	5A	6A	13A	17A	20A	25A	13A (a)
Component, wt %							
-343 °C(-650 °F)	4.3(b)	5.2(b)	trace	0.9(b)	0.0	4.2(b)	trace
343 °C x 524 °C (650 °F x 975 °F)	6.7	28.7(c)	15.7	20.7	19.1	28 5	16.3
THF-Soluble 524 °C+ (975 °F+)	52.5	16.4	31.9	24.9	27.8	16.0	39.7
IOM	28.8	39.0	41.1	45.3	43.6	41.6	36.1
Ash	6.8	8.9	6.9	7.8	7.8	8.2	5.0

(a) Date/period in question
(b) May have contained an unknown amount of water
(c) End point was ca.529 °C (985 °F), instead of 524 °C (975 °F)

Actual distillation conditions were 5 torr/348 °F pot/378 °F head (176 °C pot/192 °C head) and 5 torr/608 °F pot/638 °F head (320 °C pot/337 °C head) to provide effective cut points of 343 °C (650 °F) and 524 °C (975 °F).

TABLE 3

ASH ELEMENTAL COMPOSITION OF PRESSURE-FILTER CAKES FROM HTI RUN ALC-1

	Period 6 (Toluene-extracted)	Period 13 (Unextracted)	Period 17 (Unextracted)	Period 20 (Unextracted)	Period 25 (Unextracted)
Moisture, wt % As-Determined	0.30	0.23	0.31	0.43	0.41
Ash, wt % MF, Including SO_3	70.45	47.49	45.24	45.61	45.20
Ash, wt % MF, SO_3-Free	51.58	42.60	40.58	41.19	31.64
Major Ash Elements, Oxide wt % of Ash					
MoO_3	0.21	0.31	0.49	0.56	0.21
Na_2O	1.17	0.37	0.37	0.32	1.17
K_2O	0.29	0.33	0.41	0.37	0.27
CaO	14.76	6.47	6.86	6.52	17.45
MgO	3.29	1.14	1.14	1.02	3.58
Fe_2O_3	22.27	36.00	30.45	26.93	17.94
TiO_2	0.77	1.42	1.58	1.61	1.02
P_2O_5	0.81	1.18	1.33	1.33	1.00
SiO_2	19.03	27.76	30.60	32.05	15.24
Al_2O_3	10.34	15.11	16.76	17.97	11.56
SO_3	26.78	10.29	10.31	8.68	29.99
Total	99.72	100.36	100.30	96.36	99.42
Mo wt % of SO3-free ash	0.22	0.26	0.41	0.47	0.23
Fe wt % of SO3-free ash	21.27	28.07	23.75	20.85	17.92
Mo wt % of SO3-containing ash	0.16	0.23	0.37	0.42	0.16
Fe wt % of SO3-containing ash	15.58	25.18	21.30	18.84	12.55
HTI Analyses, wt % of ASTM Ash (a)					
Mo - Second Stage Sample	0.195	0.232	0.400	0.403	0.190
Fe - Second Stage Sample	9.226	21.096	18.766	17.372	13.206
Mo - First Stage Sample	0.198	0.188	0.218	0.232	0.162
Fe - First Stage Sample	13.316	17.468	12.721	12.202	10.708

(a) Source: Table 16 (page 40) of the HTI draft Run ALC-1 report of June 1996.

TABLE 4

ASH ELEMENTAL COMPOSITION OF FEED SLURRY 975 °F⁺ RESIDS FROM HTI RUN ALC-1

	Period 5	Period 6	Period 13	Period 17	Period 20	Period 25 (a)
Moisture, wt % As-Determined	0.26	2.14	1.48	1.25	0.96	2.16
Ash, wt % MF, Including SO_3	8.12	15.26	10.29	10.18	10.02	13.20
Ash, wt % MF, SO_3-Free	6.06	11.42	9.27	9.26	9.16	9.90
Major Ash Elements, Oxide wt % of Ash						
MoO_3	0.20	0.20	0.43	0.48	0.56	0.20
Na_2O	1.15	1.17	0.43	0.38	0.31	1.18
K_2O	0.29	0.29	0.33	0.39	0.36	0.28
CaO	14.53	14.41	6.88	6.57	6.14	16.44
MgO	3.16	3.18	1.25	1.09	0.98	3.43
Fe_2O_3	23.13	23.64	33.69	30.29	29.37	19.17
TiO_2	0.79	0.78	1.41	1.59	1.57	1.02
P_2O_5	0.60	0.62	1.16	1.31	1.27	0.99
SiO_2	19.76	19.06	28.28	30.92	31.64	19.63
Al_2O_3	10.73	11.04	15.84	17.38	17.74	12.37
SO_3	25.41	25.16	9.90	9.03	8.60	25.01
Total	99.95	99.77	99.64	99.43	98.54	99.72
Mo wt % of SO_3-free ash	0.20	0.20	0.36	0.40	0.46	0.20
Fe wt % of SO_3-free ash	21.69	22.09	26.11	23.29	22.47	17.68
Mo wt % of SO_3-containing ash	0.15	0.15	0.32	0.36	0.42	0.14
Fe wt % of SO_3-containing ash	16.18	16.53	23.52	21.19	20.54	13.41

(a) Single determination, others are averages of duplicate determinations.

TABLE 5

MO AND FE BALANCE AND APPARENT ADDITION RATES FOR HTI RUN ALC-1

TOTAL Mo & Fe in Feed Slurry , (Based on coal ash and recycle IOM+ash rates):

Period	MF Coal In, g	Fe_2O_3, g	Coal Ash In, g	Coal + Cat Ash In, g	Recycle Ash In, g	Total Ash In, g	Total Mo In, g	Total Fe In, g
6	32,405	454	1,782	2,236	4,439	6,675	13.35	1,475
13	26,786	375	884	1,259	3,536	4,795	17.26	1,252
17	26,365	258	870	1,128	3,691	4,820	19.28	1,122
20	20,078	197	663	859	2,570	3,429	15.77	771
25	22,362	219	1,230	1,449	2,303	3,752	7.50	671

Mo & Fe in TOTAL PFC OUT:

Period	Tot. PFC Out, g	PFC Ash Out, g	PFC Mo Out, g	PFC Fe Out, g	Mo Out/In, Ratio	Fe Out/In, Ratio		
6	13,215	6,816	15.00	1,450	1.12	0.98		
13	12,537	5,341	13.89	1,499	0.80	1.20		
17	11,775	4,778	19.59	1,135	1.02	1.01		
20	8,919	3,674	17.27	766	1.09	0.99		
25	12,307	3,894	8.96	698	1.19	1.04		

Mo & Fe from RECYCLE PFC:

Period	PFC Recycle, g	PFC Ash Recycle, g	PFC Mo Recycle, g	PFC Fe Recycle, g	Mo Rec/In, Ratio	Fe Rec/In,		
6	8,638	4,455	9.80	948	0.73	0.64		
13	9,084	3,870	10.06	1,086	0.58	0.87		
17	9,030	3,664	15.02	870	0.78	0.78		
20	6,618	2,726	12.81	568	0.81	0.74		
25	7,420	2,348	5.40	421	0.72	0.63		

Mo & Fe NET in Fresh Feed:

Period	Total Mo In, g	Total Fe In, g	PFC Mo Recycle, g	PFC Fe Recycle, g	Calc. Fresh Mo In, g	Calc. Fresh Fe In, g	Calc. Fresh Mo Rate,	Target Mo Rate, ppm
6	13.35	1,475	9.80	948	3.55	527	110	100
13	17.26	1,252	10.06	1,086	7.20	166	269	100
17	19.28	1,122	15.02	870	4.25	252	161	70
20	15.77	771	12.81	568	2.96	202	148	70
25	7.50	671	5.40	421	2.11	250	94	70

Period	Calc Fe Total Rate, ppm	Fe from Coal, ppm	Calc. Fresh Added Fe Rate, ppm	Target Added Fe Rate, ppm				
6	16,261	2,618	13,642	10,000				
13	6,184	2,256	3,928	10,000				
17	9,565	2,256	7,309	7,000				
20	10,071	2,256	7,815	7,000				
25	11,189	2,618	8,571	7,000				

All ppm values are on a ppm (mg/kg) MF coal basis.

TABLE 6

WAX CONTENT IN VSOH
RUN ALC-1

Stream	Period	Wax Yield at Temperature (a), %		
		-5 °C	-20 °C	-35 °C
O-6 Bottoms Distillate	20	5.2	5.9	6.1
VSOH	25	1.2	1.5	2.6
VSOH, dewaxed	25	0.1	0.1	0.2

(a) Yield of wax obtained in laboratory dewaxing test with acetone at the specified temperature.

TABLE 7

NMR ANALYSES AND MICROAUTOCLAVE TESTS RELEVANT TO RUN ALC-1 DEWAXING OPERATIONS

	Period	Proton Distribution %							Microautoclave Coal Conversion, wt % MAF	
		Cond Arom	Uncond Arom	Cyclic Alpha	Alkyl Alpha	Cyclic Beta	Alkyl Beta	Gamma	Test A (a)	Test B (b)
O-6 bottoms, 454 °C⁻ (850 °F⁻) distillate	13	15.8	9.9	15.8	11.1	12.6	23.8	10.8	88.1	-
O-6 bottoms, 454 °C⁻ (850 °F⁻) distillate	17	15.4	10.1	15.6	10.7	12.5	24.5	11.2	87.6	-
O-6 bottoms, 454 °C⁻ (850 °F⁻) distillate	20	16.0	10.0	16.2	11.2	12.8	23.3	10.5	86.7	78.3
O-6 bottoms, 454 °C⁻ (850 °F⁻) distillate	25	14.3	9.2	14.8	11.3	15.4	21.2	13.8	88.4	79.4
VSOH	13	12.3	10.5	15.5	12.5	14.1	23.9	11.1	-	-
VSOH	17	13.1	10.3	14.9	11.5	13.1	24.9	12.3	-	-
VSOH, 343-399 °C (650-750 °F)	20	15.3	8.6	16.3	10.8	12.9	25.9	10.2	-	-
VSOH, DW-HT, 343 °C⁺ (650 °F⁺)	21A	9.0	6.6	15.3	10.5	19.8	23.4	15.3	92.7	-
VSOH, IBP- 524 °C (975 °F)	25	17.8	6.2	17.8	11.0	15.7	20.8	10.6	89.5	80.9
VSOH, DW	25	17.8	9.8	17.1	11.3	14.5	18.0	11.5	89.7	82.3
VSOH, DW-HT	25	8.2	6.1	15.8	9.9	21.4	23.1	15.5	92.6	89.9
VSOH, DW-HT, 343 °C⁺ (650 °F⁺)	25	10.8	6.3	18.4	10.6	19.7	21.3	13.0	94.4	90.5

(a) Modified equilibrium test, Old Ben Mine coal

(b) Black Thunder Mine coal (4.55 g), solvent (5.45 g), 30 min, 440 °C (824 °F), 10.3 MPa (1500 psig) H_2 (cold)

TABLE 6

SUMMARY OF H-NMR ANALYSES AND MICROAUTOCLAVE TESTS RELEVANT TO ALC-1 DEWAXING OPERATIONS

Period	Stream	^{1}H-NMR Proton Types, %			Microautoclave Coal Conversion, % MAF	
		Aromatic	Cyclic	Paraffinic	Test A (a)	Test B (b)
13,17, 20	VSOH/O-6 Bottoms Distillate	24.5 ±1.4	28.7 ±0 6	35.4 ±1.2	87.4 ±0.7	78.3
25	VSOH/O-6 Bottoms Distillate	23 7 ±0 3	31.6 ±1.7	33.4 ±1.4	89.0 ±0 8	79.4/80.9
25	VSOH, dewaxed	27.6	31.6	29.5	89.7	82.3
25	VSOH, dewaxed and hydrotreated	14.3	37.2	38.6	92.6	89.9
25	VSOH, dewaxed and hydrotreated, 343 °C+ (650 °F+)	17.1	38.1	34.3	94.4	90.5

(a) Modified equilibrium tests with Old Ben Mine Coal
(b) Black Thunder Mine coal (4.55 g), solvent (5.45 g), 30 min, 440 °C (824 °F), 10 MPa (1500 psig) H_2 (cold)

TABLE 9

ANALYSIS AND DE-OILING YIELDS OF WAX FROM RUN ALC-1, Period 25

	wt % (a)
Elemental Analysis of Original Wax	
C	73.82 (b)
H	13.70
N	0.20
S	0.06

(a) Average of four determinations. Repeatability was ca. ±5% relative.
(b) Suspect low, cf. text.

Yields from De-Oiling Original Wax	
Fraction	wt %
De-Oiled Wax	74.7
Oil	19.4
Insolubles	1.7
Losses	4.2

Proton Distributions of De-Oiling Fractions							
	Proton Distribution, %						
	Cond Arom	Uncond Arom	Cyclic Alpha	Alkyl Alpha	Cyclic Beta	Alkyl Beta	Gamma
Original Wax	2.5	1.4	4.6	3.2	8.1	68.7	11.5
De-Oiled Wax	0.5	0.2	0.6	0.7	3.3	80.4	14.4
Oil	8.9	3.3	8.5	5.5	10.6	49.9	13.3